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SUBTITLE: Transport and Fate of Nitroaromatic and Nitramine
            Explosives in Soils From Open Burning/Open Detonation
            Operations: Radford Army Ammunition Plant (RAAP)

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CONTRACTING
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RESEARCH, DEVELOPMENT & ENGINEERING CENTER

U.S. ARMY CHEMICAL AND BIOLOGICAL DEFENSE COMMAND

ERDEC-TR-133

TRANSPORT AND FATE OF NITROAROMATIC AND NITRAMINE EXPLOSIVES IN SOILS FROM OPEN BURNING/OPEN DETONATION OPERATIONS:

RADFORD ARMY AMMUNITION PLANT (RAAP)

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13. ABSTRACT (Maximum 200 words) This report describes soil column experiments (task 1) used to study fate and transport of OB/OD residues within the upper 1 m of soil. [Terrestrial (task 2) and aqueous (task 3) toxicity studies are reported elsewhere.] Intact soil-cores were collected from Radford Army Ammunition Plant. Intact soil-cores were also collected from Anniston Army Depot, Milan Army Ammunition Plant, and Pueblo Army Depot; results of these three studies are reported elsewhere. Soil columns were housed in controlled-environment chambers, and each soil column was formulated to approximate the major OB/OD residues found at the respective sites. Synthetic rainwater was added to the columns twice weekly, and a controlled tension applied. Leachates were collected biweekly. Columns were analyzed at 3-month intervals through 9 months. Columns were cut into 2.5-cm transverse sections, and subsamples were air-dried, ground, and extracted. Extracts and leachates were analyzed by HPLC for explosives to reveal fate and transport behavior. Leachates contained no munition residues or transformation products (TP). TNT, TNB (TP), 2,4-DNT, 2,6-DNT 2-amino-DNT (TP), and 4-amino DNT (TP) all migrated vertically but <7.5 cm (3") deep into the soil. Due to weathering, TNT, TNB, 2,4-DNT, and 2,6-DNT became bound within the soil A horizon.

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Munitions	2,4-DNT	4-Amino-DNT	16. PRICE CODE
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Munition-contaminated soil

PREFACE

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CONTENTS

List of Figures

LIST OF TABLES

7

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TRANSPORT AND FATE OF NITROAROMATIC AND NITRAMINE EXPLOSIVES IN SOILS FROM OPEN BURNING/OPEN DETONATION OPERATIONS:

RADFORD ARMY AMMUNITION PLANT (RAAD)

1.   INTRODUCTION

       a. Out-of-date and out-of-specification munitions have commonly been disposed of by burning, or by detonation, on unprotected ground.[1] Through the promulgation of various environmental regulations, this practice has recently been limited. Burning pans and closed treatment systems have been used at various installations to mitigate environmental contamination. However, questions concerning the transport and transformation of open burning/open detonation (OB/OD) ash and waste explosives in soils and their environmental toxicity needed to be answered (AEHA, 1986).[2]

       The standard practice of OB/OD of munitions historically involved quantities of explosives up to thirty tons per disposal event, and generated a mixture of contaminants into the immediate area at high concentration.[3] At many military installations OB/OD sites consist of multiple disposal areas. These OB/OD sites number in the hundreds, and have been developed and used by both the military and their civilian contractors during much of this century. Many of these sites have records inadequate to predict the nature and extent of the contamination. Residue from OB/OD contains both burned and unburned explosives, but environmental weathering and microbial action are known to produce modifications of these compounds.[4,5,6] Estimation of the environmental impact of OB/OD contamination at an individual site requires detailed knowledge of the type and amount of the chemical contaminants present and an understanding of their migration behavior within the soil.

       The purpose of this project was to:
1) determine the transport and transformation of OB/OD contaminants in soil, 2) measure the toxicity of soils contaminated with explosives and 3) measure the toxicity of soil leachates. Three tasks were conducted to address the goals of the program. The first task used intact soil columns to measure the transport and transformation of chemicals in OB/OD ash and explosives of concern. The other two tasks involved determining the toxicity of explosives in soil to earthworms, and the toxicity of aqueous soil extracts to *Daphnia magna*.

       In task one, intact soil cores were collected from Radford Army Ammunition Plant (RAAP), Virginia; Milan Army Ammunition Plant (MAAP), Tennessee; Pueblo Army Depot (PAD),

Colorado; and Anniston Army Depot (AAD), Alabama. The predominant explosives at each site were monitored in their respective soil-core columns for transport and transformation in the soil. Breakthrough and subsequent concentrations of the chemicals in the leachates collected from the columns were determined. Chemical transport and transformation experiments involved leaching soil columns with synthetic rainwater for up to 243 days. This report presents the data for Radford Army Ammunition Plant soils.

In task two, standard 14-day earthworm toxicity tests were conducted on OB/OD residues and specific explosives (results reported separately, in another technical report entitled **Toxicity of Selected Munitions and Munition-Contaminated Soil to the Earthworm** *Eisenia foetida*).[7] In task 3, soil/water extracts were prepared, to partition water soluble biologically available components from the soil. These aqueous extracts were tested for toxicity to the aquatic organism *D. magna* (results reported separately, in another technical report entitled **Determination of Soil Toxicity to** *Daphnia magna* **Using an Adapted Toxicity Characteristic Leaching Procedure**).[8] The sensitivity of the *D. magna* method makes it a useful tool in assessing the impacts of contaminated soils. The results of this project will support site closure assessments at OB/OD sites, answer critical questions on the transport of explosives in soil, and address environmental toxicity data gaps.

In task one intact soil-core columns were collected on-site to study the transport and transformation of munition residues in site-specific soils. Intact soil-core columns were collected rather than collecting bulk samples of soil for packed-column studies because soil physical and chemical characteristics are typically, sometimes dramatically, altered by the drying, sieving, and storing of soils necessary for preparing packed columns. Furthermore, such handling may also cause inappropriate and radical change in the ability of soil to degrade xenobiotics[9] or utilize naturally occurring compounds.[10] Intact soil cores offer the potential for a realistic view of site-specific soil conditions as they exist in the field, yet are portable so they may be studied closely in the laboratory under conditions that simulate those occurring in the field. If appropriate precautions are taken during the collection, transport, and study of intact soil cores, information obtained for site-specific soil conditions may also give added insight to the processes controling the transport and transformation of munition residues in soils. Many investigators acknowledge the advantages of using intact soil cores for study, but apply methods that require at least one transfer of the soil core from the collection probe to its destination column, potentially causing disruption of the soil core and alteration of its characteristics. However, a group of scientists[11,12] have developed a system for taking intact soil cores, and have applied

10

the system to the extent that it was accepted as a standard
method for soil microcosm research by the U.S. Environmental
Protection Agency[13] and the American Society for Testing and
Materials.[14] The system used during the investigations detailed
in this report is an adaptation of those soil microcosm methods,
with various refinements to more realistically assess the
transport and transformation of chemicals in soils.[15] The
methods presented the following section (II. Soil Methodology)
describe these improved methods for 1) taking and directly
delivering soil cores into their respective columns with minimal
disturbance of the soil sample; and for 2) controlling
environmental parameters of the soil cores during study including
soil temperature and moisture regime, including quantity,
quality, and intensity of simulated rainfall. These factors
directly impact on the chemical, physical, and biological
properties of the soil, and potentially affect the resulting
transport and degradation of chemicals within soil[16] and their
toxicity.[17]

        RAAP was selected as the first site for
collection of samples, characterization, and investigation. RAAP
has an open burning area, and has burned waste explosives from
their manufacturing operations containing 2,4,6-trinitrotoluene
(TNT), 2,4-dinitrotoluene (2,4-DNT), and 2,6-dinitrotoluene
(2,6-DNT). Burning operations were carried out in burning pans,
however contamination of the soil did occur.

11

a. Collection of Intact Soil Cores

Prior to initiating collection of soil cores, a visual inspection of the OB/OD site was performed to ensure that the soil types conformed to those specified in the soil survey maps, obtained from the U.S. Soil Conservation Service.[18] Next a site of the same soil type and characteristic as that of the OB/OD area was located. In order to be selected, a site must be free from contamination by munition residues, preferably undisturbed, and have an area large enough that sampling near soil-type transition areas or obvious physical discontinuities was avoided.

In the field prior to sampling on-site, the soil was brought to field moisture capacity. Watering of the soil was initiated at least 24h before sampling to ensure sufficient time for both wetting, and drainage of excess water. A sampling grid was then layed out at the site selected so soil-cores would be taken every 4 feet, on center. This was done to ensure that there was sufficient work area around each sampling location to prevent compaction of adjacent locations during sampling. Each site was measured and sampling locations were marked with flags. Native vegetation (primarily grasses) were cut at the soil surface and the aerial portions of the cut plants were removed prior to sampling the soil.

The probe (Fig. 2.1) was lifted into the air and moved to each sampling location using the front-end loader and a chain. An aluminum stop-plate, 18" x 18" x 0.5" (45 cm x 45 cm x 1.3 cm) with a central hole for locating the probe, was placed over the sampling location prior to pushing the probe into the soil. The stop-plate allowed more uniform samples to be taken. A total of thirty soil-core samples were taken per site to ensure an excess of available columns[19] from which to initially test and ultimately select the final twelve columns per study. The soil probe was pushed rather than pounded into the soil to alleviate zonal compaction and minimize disruption of the soil being taken.[20] To prevent distubance of the soil at adjacent sampling locations, the front-end loader was brought in perpendicular to the area in its approach to the first sampling location; after the sample was taken, the loader was backed out, moved to the right, again moved in perpendicular to the next sampling location; and this process continued until sufficient soil-core columns had been collected.

For the soil that entered the probe during collection of intact cores, the maximum clearance discrepancy allowed (using the tolerances specified, Fig. 2.1) during delivery of soil into the high density polyethylene (HDPE) pipe

FIGURE 2.1   CROSS-SECTION OF SOIL SAMPLING PROBE WITH SOIL-CORE
ENCASED IN HDPE.

CLEVIS; FOR
HYDRAULICS.

CAP, COLD-ROLLED
STEEL; 2.5 cm THICK
AT TOP, 1.3 cm WALLS,
11.5 cm ID.

PRESSURE RELIEF
PORTS (2).

HEAD SPACE, 10 cm.

ALLEN HEAD CAP
SCREWS (6);
0.4 cm DIAM.,
IN TWIN
2.5 cm X 2.5 cm
FLANGES.

SHAFT, HOT-ROLLED STEEL
TUBING; 1.3 cm WALLS,
11.5 cm ID FLANGE TO
SHOULDER, 10.3 cm ID
SHOULDER TO CUTTING TIP.

PIPE, BLACK, HDPE,
ENCASES SOIL-CORE;
86 cm, 11.5 cm OD,
10.3 cm ID.

HDPE PIPE CONTAINING
SOIL-CORE; REPLACEMENT
HDPE PIPE ACQUIRES
NEXT SOIL-CORE.

SOIL-CORE: 6300 cm^3 SOIL max.
(76 cm max. x 10.3 cm DIAM.),
DIRECTLY ENTERS
HDPE PIPE.

SHAFT, WITH FEMALE
THREADS.

TIP, D2 TOOL STEEL
HARDENED TO ROCKWELL
C-SCALE 37-39;
14' TAPER OD, 10.3 cm ID.

TIP, WITH MALE THREADS.

13

inside the probe was <0.05-cm, resulting in a soil-core diameter of 10.3-cm ±<0.1. The HDPE pipe used in this study was opaque, the grade and quality used in high pressure gas pipelines. HDPE pipe was purchased in 12.2-m (40-ft) lengths, and prior to going to the field was cut and sanded to the specified dimensions. The HDPE pipe collection tubes were inert hydrophobic barriers that remained an integral part of the soil-core columns. Thus, disruption of the soil due to column-to-column transfers was eliminated. Upon removal of the HDPE collection tube containing the soil-core from the probe, measurements were taken of the resulting head space within each column; additionlly it was advantageous to measure the depth of soil penetration by the probe that results from sampling. If dramatic inconsistencies occurred in the depth values in the field, the corresponding columns were rejected and others taken in their place. After removal from the probe, each HDPE collection tube containing a soil core was immediately placed in a set of "V" blocks for sealing and packaging. Each end of the HDPE collection tube was sealed with a barrier-cap consisting of double layers of 4-mil thick polyethylene sheeting, then sealed with duct tape to the HDPE pipe. This minimized gas exchange and prevented moisture loss from the soil cores. A sufficient supply of barrier-caps were prefabricated in the laboratory, prior to going to the sampling site, in order to decrease the amount of field time required to seal a soil-core sample tube. Barrier-caps were prefabricated by cutting out a 10" square piece of double-layered (2 x 4-mil) polyethylene sheeting, centering the square over an empty HDPE collection tube, and wrapping it around while pushing it down over the tube. This wrap was then held in place by a thick rubber band so a piece of duct tape could be placed tightly around the wrap 1" (2.5 cm) from the end of the HDPE collection tube. The corners of the square wrap (excess) were then cut off around the tube 2" (5.0 cm) below the tape. When using these barrier-caps in the field, the barrier-cap is slipped onto the end of the HDPE collection tube and an additional piece of duct tape is used to completely seal the edge of each barrier-cap to the outer surface of the tube. After the ends were sealed, each tube was labeled with the date, location, and collection site number.

Collected soil cores in their HDPE tubes were placed into 32-gal (120-L) opaque polyethylene containers, which contained a 6" (15 cm) thick foam rubber pad in the bottom. A group of HDPE tubes were placed on the pad in each container with the soil end down. The sealed columns extended out of the top of the containers, and through the container covers which had been cut to fit the columns. Black polyethylene plastic bags were used to cover the tops of the sealed columns. All soil samples obtained from a site were transported back to the laboratory upright in padded containers to minimize disruption of the soil cores during transport.

b.   Soil Column Preparation and Testing

Afterward in the laboratory, selected soil-core columns were trimmed of excess soil if any was present, fitted with a porous ceramic disk (2.5 um pores) in opaque HDPE endcaps containing fittings for teflon tubing with in-line monitoring and shut-off valves (Fig. 2.2).   The HDPE end-caps used in this study were the grade and quality used in high pressure gas pipelines, however prior to use each was milled to contain a well for the controlled-pore ceramic plate, then milled again and threaded for tubing fittings.   End-cap fittings were also HDPE.   The intact soil-core columns were then transferred into the controlled temperature (controlled environment soil-core microcosm unit; CESMU) chamber (Fig. 2.3).   The CESMU chamber was housed in a greenhouse for high-temperature control, and was equipped with 10.5 MJ h^{-1} cooling capacity sufficient for maintaining a constant temperature within entire soil columns for isothermic studies at 25.0 ± 0.1 °C.   During these investigations the tops of the columns were left open to receive sunlight, sufficient for plant growth (however, they could instead be covered with an opaque insulated cover spanning all columns to eliminate photodegradation processes).   Controlled tension (vacuum) was applied equally at the bottom of each soil column across the controlled-pore ceramic plate, at 30-35 kPa; tension was regulated and monitored.

The tension that was applied is comparable to that encountered in the field as a result of combined soil matric and gravitational forces; thus avoided were undue flooding, the buildup of a hanging column of water in the lower portion of columns, and artificial changes in soil redox potential in response to steady-state alteration of the soil water content, as can happen when gravitational forces alone are relied upon to promote water flow through soil columns.   Before initiating any studies of the fate, migration, and degradation of munition residues, the soil-core columns in the CESMU chamber were saturated with water and equilibrated under tension (48h minimum), after which water thru-put was evaluated for each of the initially selected columns.

The initial selection of twelve columns per soil type (site) for preliminary testing was done on the basis of similarity of head space within columns, an easily obtained measurement that is the compliment to column length.   Using the sampling methods and measurements described above, a group of columns differing in length by only centimeters (Fig. 2.4) was obtained that provided a sufficient number of columns from which to select those for the preliminary testing of water flow (thru-put).   Soil-core columns were initially selected on the basis of similarity of length; and replacement columns within each soil type group, if needed, were those with the next closest

15

FIGURE 2.2   SOIL-CORE COLUMN INCLUDING END-CAP AND FITTINGS.

PIPE, BLACK
POLYETHYLENE
(HDPE), ENCASES
SOIL-CORE;
85 cm, 11.5 cm OD,
10.3 cm ID.

SOIL-CORE,
6300 cm^3 SOIL max.;
76 cm max.x 10.3 cm DIAM.

END CAP, HDPE;
WITH FITTINGS.

TEFLON TUBING;
WITH THREE-WAY VALVE/
VACUUM MONITOR.

CERAMIC PLATE,
2.5 μm PORES;
1.0 cm x 10.5 cm DIAM.

POLYETHYLENE
BUNG.

SILICONE SEALANT.

TO VACUUM
AND ACCESSORIES.

SILICONE RUBBER
GASKETS.

ONE-WAY VALVE
N$_2$ (PURGE GAS).

SAMPLE COLLECTION FLASK (1-L);
210 mL EQUIVALENT TO 2.5 cm
COLUMN THROUGH-PUT.

FIGURE 2.3 CROSS-SECTION OF CESMU SYSTEM SHOWING ONE SOIL-CORE COLUMN AND VACUUM SYSTEM.

TEFLON TUBING.

CHAMBER (PARTIAL)
CROSS-SECTION,
TEMPERTURE
CONTROLLED (WITH
FOAM-FILLED
10 cm WALLS);
INTERNAL DIMENSIONS
(cm) 84 WIDE x 210
LONG X 84 HIGH
ABOVE GRILLE,
AND 18 BELOW.

COLLECTION
FLASK. 1-L.

GRILLE.

PVC TUBING.

MULTIPORT SEQUENCING VALVE;
SUPPORTS 12 SOIL-CORE COLUMNS.

IMPROVEMENT FEATURE
FOR CESMU SYSTEM.

VACUUM
REGULATOR.

SAFETY
OVERFLOW
FLASK.

VACUUM PUMP
DUAL CYLINDER.

VACUUM STORAGE TANK, 38-L.

FIGURE 2.4   FREQUENCIES OF SOIL-CORE COLUMN DEPTHS: WHEELING
             SANDY LOAM SOIL (RAAP).
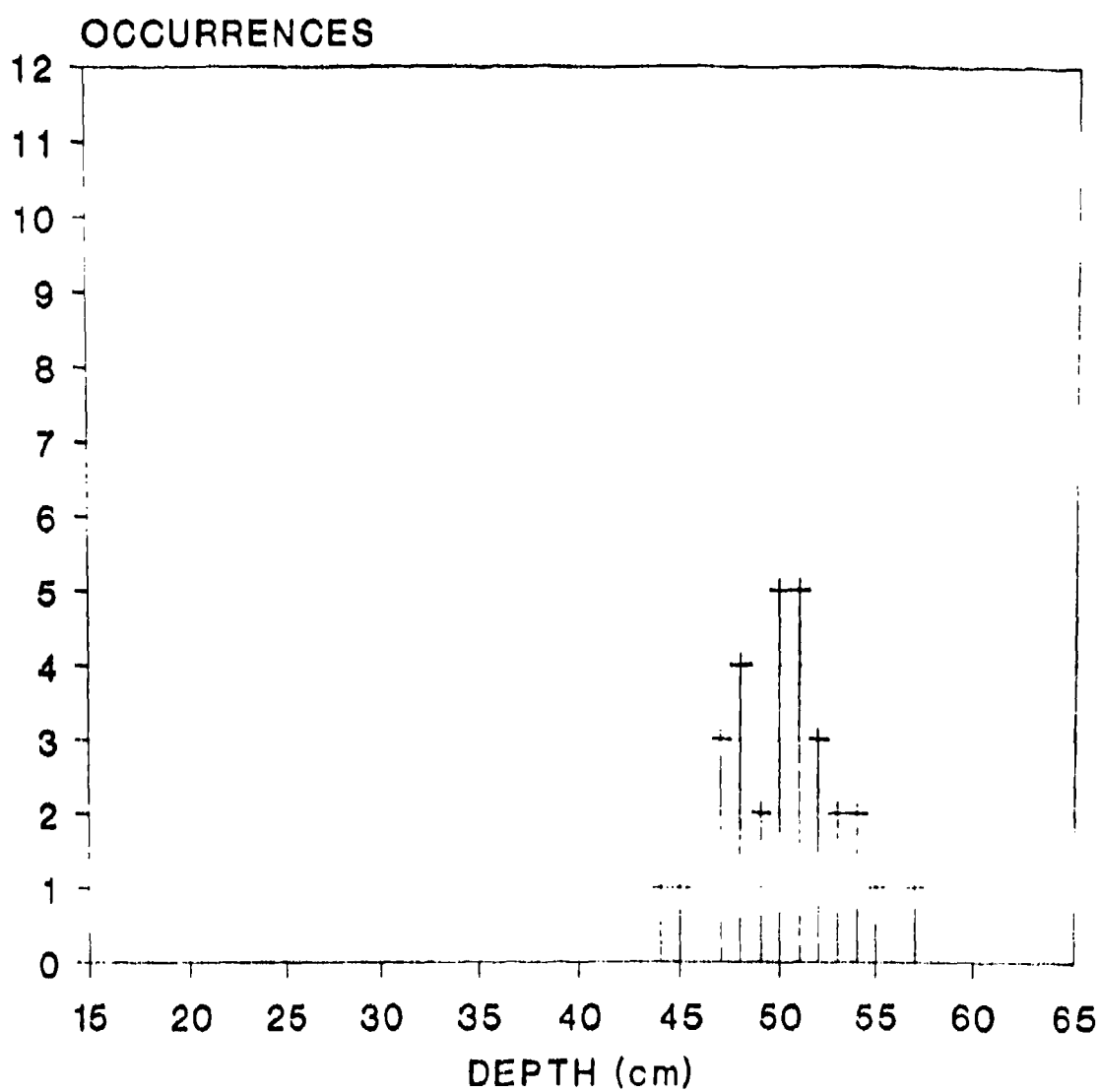
to the mean length. For the initially selected columns that were
found to have rates of flow or water thru-put substantially
different than the median, replacement columns were selected, and
then similarly evaluated. Outlier-columns within each soil type
(based on values of water thru-put, when water was applied,
monitored, and sampled analogous to artificial rain additions
described below) were replaced until the standard deviation about
the mean value for water thru-put was $\leq 10\%$. Then, based on the
adjusted mean excluding outliers, any additional columns with
thru-put values falling outside of the adjusted mean $\pm$original
standard deviation were also replaced, until all test columns
fell within one standard deviation of the mean. Representative
columns were thus identified and retained for study in the CESMU
chamber.

c. Spiking of Soil Columns

OB/OD contaminated soil was collected from an
open detonation pit that had the most recent disposal operation.
This contaminated PAD soil was air-dried, extraneous materials
(nails, stones, etc.) removed, crushed, and ground to pass a 2-mm
nylon seive. After this, the type and quantity of munition
residues was determined.   Then a mixture of the prepared
detonation pit soil and explosives, related to munition residues
detected in the screening analysis, was prepared.

Soil contaminated with residues from OB/OD
activities was collected, and the type and quantity of munition
residues determined.   A mixture of the native soil and the type
of munition residues detected in the screening analysis was
prepared.   After twelve representative soil columns collected
from the site were identified and randomly placed in the CESMU
according to the specifications in this report, the mixture of
soil and explosives was added atop the soil surface of the
randomly assigned treatment columns. Thus each third of the
CESMU chamber contained a randomized complete treatment block,
consisting of three treatment columns and a control column.
During preparation of the mixture, 125.0 mg kg^{-1} 2,4-DNT and 40
mg kg^{-1} 2,6-DNT were added, while ambient concentrations of TNT
and TNB were used.   The spiking mixture was then analyzed and
determined to contain the following concentrations of
acetonitrile extractable explosives and transformation products
(mg kg^{-1}):   61.7 $\pm$7.5 TNT, 103.8 $\pm$1.0 TNB, 115.8 $\pm$0.2 2,4-DNT and
36.6 $\pm$0.8 2,6-DNT. Each of the 9 treatment soil columns from the
RAAP site received a mass of soil equivalent to 1" (2.5 cm) of A
horizon soil (yielding approximately 210 mL of the mixture, after
settling), while control columns received the same mass of
uncontaminated soil from the site.

One inch (2.5 cm) synthetic rainwater
(pH 4.60 $\pm$0.02; constituents detailed in this report was
administered at the top to the center of each soil-core column

twice a week at the rate of 1" h^{-1} (7 ums^{-1}) using a peristaltic pump. Resulting leachates were collected into vacuum flasks and kept at soil column temperature (25.0 °C). Leachates were harvested bi-weekly, and analyzed for munition residues and transformation products; the pH of leachates was determined at the time of collection. The maximum duration of leaching was 39 weeks.

Harvest of Soil Columns

Four soil-core columns (three treatment and one control) were harvested after each designated leaching interval. Harvesting of columns occurred after 13, 26, and 39 weeks of leaching, respectively. Column harvest, sectioning, and preparation for analyses, are described in this report.

d. Analytical Methods

The analytical methods and procedures for determining munition residue concentrations in leachates were the same as described in Section 3 of this report, with the following exceptions:

i. The extracts of soil sub-samples were filtered through a Gelman 0.45 um Acrodisc-CR disposable filter.

ii. Sub-samples of the aqueous leachates were adjusted to pH 6.00 ±0.05 and made to contain 300 g L^{-1} NaCl. Two hundred mL of the resultant solution was put through a J.T. Baker 40 um Sep-Pak Octadecyl (C18) disposable cartridge at a rate of 1.8 mL min^{-1}. Cartridges were prepared for use by wetting with 2 mL methanol, followed by 2 mL deionized water. Munition residues were eluted from the cartridges with 2x 1-mL additions of methanol, and eluates were analyzed by HPLC.

Criteria of detection values for treatment soil sampless for each explosive and their transfor-mation products, including details of calculation, are given in Appendix B.

e. Simulated Rainfall and Resulting Leachates

In the laboratory, synthetic rainwater was formulated based on records of the constituents of rainfall across Pennsylvania,[21,22,23] and used to represent the constituents and characteristics of rainfall in the mid-Atlantic coastal region. The constituents of the synthetic rainwater were (uM, in deionized water) 15 SO_4, 11 NO_3, 9 Cl, 25 NH_4, 7 Ca, 3 Mg, 3 Na, and 2 K; pH was adjusted to 4.60 ±0.02 using a 1.35:1 mixture of 1M H_2SO_4 and 1M HNO_3. Synthetic rain was administered at the top to the center of each soil-core column twice a week at the rate of 1" h^{-1} (7 um s^{-1}) using a peristaltic pump.

20

Resulting leachates were collected via teflon tubing into 1-L flasks in darkness, and kept at soil column temperature (25.0 $\pm$0.1 °C) inside CESMU until removed for analyses. Nitrogen gas was provided for purging the end-cap, but only during collection of the aqueous leachates. Leachates were harvested at regular intervals, and analyzed for munition residues and transformation products; the pH of leachates was determined upon collection using a combination pH electrode and digital pH multimeter.

Synthetic rainwater (pH 4.60 $\pm$0.02) in the amount of 0.2" (0.6 cm) was administered at the top to the center of each soil-core column twice a week at the rate of 1" h^{-1} (7 um s^{-1}) using a peristaltic pump. Resulting leachates were collected into vacuum flasks and kept at soil column temperature (25.0 °C). Leachates were harvested twice-weekly, and analyzed for munition residues and transformation products; the pH of leachates was determined at the time of collection. The maximum duration of leaching was 32.5 weeks.

f. Harvest of Soil Columns

Replicate soil columns were harvested at regular intervals following leaching, sealed (in the same manner as when collected from the field, Section 2.b), then frozen. Afterward, the frozen soil cores encased in HDPE pipe were carefully cut open using a router (with the depth of penetration set to the wall thickness of the HDPE tubes) and a hand guide, allowing the resulting intact soil core to rest in the lower half of the HDPE pipe. Soil cores were then slowly thawed in the horizontal position to effectively eliminate longitudinal migration. Then from top to bottom, the soil cores were marked into sections using a spatula to indicate 1" (2.5 cm) depth intervals. The soil was then sectioned into 1" depth x 4" diam. (2.5 cm x 10.3 cm) discs. Each disc was individually transferred into a clean polyethylene bag, air-dried, crushed, and ground to silt consistency ($\leq$150 um). Using similar sectioning methods but larger section sizes, replicate bulk density determinations were done individually for A and B horizons using the extra soil-core columns.

g. CESMU System Integrity

Although controlled tension was applied equally at the bottom of each soil-core column during studies and was regulated and monitored, the failure to maintain tension at any single column potentially affected the tension on the remaining columns until the failing column was repaired or eliminated. Generally this problem occurred only during the set-up and preliminary testing of columns, and resulted from an immediately repairable minor leakage. Infrequently, this problem occurred due to handling of system components during sampling of leachates, but again caused only minor leakage of vacuum and was easily and

21

immediately repairable.

Physical and mechanical systems supporting the CESMU chamber and rainfall delivery functioned well under almost constant use for more than two years. Over this period, the transport and transformation of munition residues were investigatied in four different site-specific soils, using twelve study columns per soil type (site), with individual studies lasting from six to nine months depending upon the lability of chemicals investigated. During these studies only one study-column failed out of fourty-eight total columns selected for investigation, and the remaining soil columns had relatively constant outputs within respective soil types.

Mechanical-part failures during this period included only one vacuum pump failure (replaced with a back-up unit while the original was rebuilt), and one vacuum regulator that failed inspection during an investigation and was immediately replaced with a back-up unit. Performance of the physical and mechanical systems was high, providing high confidence in maintenance of the conditions and limits designed for the studies.

h. Determination of Selected Soil Parameters

For this investigation several soil physical and chemical parameters were selected for determination by the University of Maryland Soil and Plant Testing Laboratory, College Park, MD. The soil properties chosen were selected to more fully characterize and understand the role of the effects of specific soil properties on the transport and transformation of munition residues, and their transformation products. Soil properties determined included percent sand, silt, clay, and organic matter, the cation exchange capacity (CEC), and soil pH.

22

3. DETERMINING MUNITION RESIDUES AND THEIR TRANSFORMATION PRODUCTS

a. Analytical Methods Development Using High Performance Liquid Chromatography (HPLC)

The quality control program for this study was based on a system that assessed sample preparation, analyte recovery, and analytical precision and accuracy. Details of this program are presented in Appendix A.

Our approach to analytical determinations supporting these investigations was based on a two step process. The first step was qualitative analysis of contaminated surface samples to screen for compounds present in environmentally significant concentrations. Due to the variety of military explosives and their environmentally modified forms, a new method was required to chromatographically isolate and thus identify the majority of the compounds likely to be encountered. The second step was quantitation of these contaminants in soil and in water that leached through this soil. Screening and quantitation processes required different HPLC methods because quantitation required greater analytical sensitivity than the screening method could provide.

Sample preparation and extraction procedures were adapted from a method developed and extensively tested by Jenkins[24,25,26]. These modified procedures entailed grinding air-dried soil samples, and extracting into acetonitrile with 18 hours of sonication at 20°C. Extracts were then centrifuged at 3900 X G for 15 min, and analyzed by HPLC. The latter portion of the sequence differs from Jenkin's method in that a step requiring mixing the acetonitrile extract with an aqueous floculating solution was eliminated, and that the internal standard 1,3-dinitrobenzene (DNB) was incorporated.

An estimation of the efficiency of extraction of each compound was obtained by doping subsamples of uncontaminated surface soil with acetonitrile containing a mixture of selected OB/OD compounds plus DNB. The soil was air-dried and extracted as above, and the efficiency of extraction was calculated from the amount of each compound recovered. Because the efficiency of extraction of the OB/OD components at our test sites was similar to that of DNB, a simplified recovery correction system was possible. All soil samples were extracted with acetonitrile containing 2.5 mg L^{-1} (ppm) of DNB as an internal standard. Observed concentrations of OB/OD components in the extraction mixture were corrected for losses of internal standard that occurred during the extraction process. Corrections were also made for any increases in concentration due to evaporation of the extraction solvent.

Aqueous leachates were directly analyzed for

23

munition residues and degradation products. These determinations were done without any preconcentration, internal standardization, or other preparation.

HPLC analyses of leachates and soil extracts were done using a Hewlett-Packard (HP) 1050 HPLC system that consisted of an autoinjector, pumping module, and UV detector. Signal integration was performed with an HP 3396A integrator. All analyses except screening tests for the presence of NG were done by UV absorbance at 244 nm. NG was determined at 220 nm.

Extracts of uncontaminated soils (background) and highly contaminated surface soils were screened by the gradient method developed for this investigation. A 15-uL sample was injected onto a 4.6 X 250 mm Rainin Microsorb C18 column with a 5 um particle size, in series with a 4.6 X 250 mm Supelcosil LC-PAH column. Elution was accomplished with a methanol:water gradient (Table 3.1).

A simpler isocratic method (developed elsewhere by Miyares and Jenkins[27]) was used to substantiate identification and to quantitate contaminants. This isocratic method entailed isocratic pumping of a mobile phase of 70.7% water, 27.8% methanol, and 1.5% tetrahydrofuran, at a flow rate of 2 mL min^{-1}

Table 3.1 HPLC Time/Gradient (Methanol:Water Mixture) for Initial Screening of Samples for a Broad Range of Munition-Related Analytes and PAHs.

Time (min)	Percent Methanol (% MeOH)
0	30
1.5	33.5
6.0	47.5
24.0	51.0
35.0	54.5
60.0	100.0
80.0	100.0

through a 25 cm x 4.6 mm Supelco LC8 column of 5 um particle

size. This procedure was modified by the addition of an acetonitrile gradient to minimize peak-broadening when amino-dinitrotoluenes (amino-DNTs) were quantitated.

b. Results of HPLC Methods Development

The above procedures have proven effective in recovering and quantitating OB/OD residues in all soils tested (Table 3.2); they have the additional advantage of being simple and reproducible. However, several shortcomings were encountered. Efforts to identify some minor components of the OB/OD soil contaminant mixture were not successful due to interferences from natural soil components. Although the majority of UV-absorbing soil components elute from reverse phase chromatography before most explosives, some elute at later retention times causing a rough baseline at high sensitivities thereby making quantitation of extremely small peaks unreliable.

Table 3.2 Efficiencies of Recovery of Selected Munitions, from Soil and Water.

	Percent Munition Recovered (%), ±s		
	From soil extracted with acetonitrile		From aqueous leachate concentrates in MeOH
Compound	doped uncontam.	doped contam.	
RDX	95 ± 1	91 ± 2	38 ± 1
HMX	99 ± 6	112 ± 4	29 ±10
TNT	107 ± 1	94 ± 9	90 ± 4
2,4-DNT	103 ± 1	110 ± 5	108 ± 7
2,6-DNT	103 ± 1	103 ± 2	104 ±20
2-Amino-DNT	100 ±<1	103 ± 1	112 ±15
4-Amino-DNT	98 ± 3	102 ± 4	137 ±40
TNB	102 + 2	114 ± 3	123 ± 3

The gradient procedure presented here effectively separated components of a mixture that included most compounds likely to be encountered during analysis of soils from OB/OD contaminated sites (Fig. 3.1). It was able to detect many

1 Nitroguanidine (NQ)
2 2,4,6-Trinitrophenol (Picric acid)
3 1-Acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX)
4 Cyclotetramethylenetetranitramine (HMX)
5 1-Acetylhexahydro-3,5-dinitro-1,3,5-triazine (TAX)
6 Cyclotrimethylenetrinitramine (RDX)
7 1,3,5-Trinitrobenzene (TNB)
8 1,3-Dinitrobenzene (DNB)
9 2,4,6-Trinitrophenylmethylnitramine (Tetryl)
0 2,4,6-Trinitrotoluene (TNT)
1 4-Amino-2,6-dinitrotoluene (4-Amino-DNT)
2 2-Amino-4,6-dinitrotoluene (2-Amino-DNT)

13 2,6-Dinitrotoluene (2,6-DNT)
14 2,4-Dinitrotoluene (2,4-DNT)
15 Naphthalene
16 Acenaphthylene
17 Fluorene
18 Phenanthrene
19 Anthracene
20 Fluoranthrene
21 Pyrene
22 Benz(a)anthracene
23 Chrysene
24 Benzo(a)pyrene
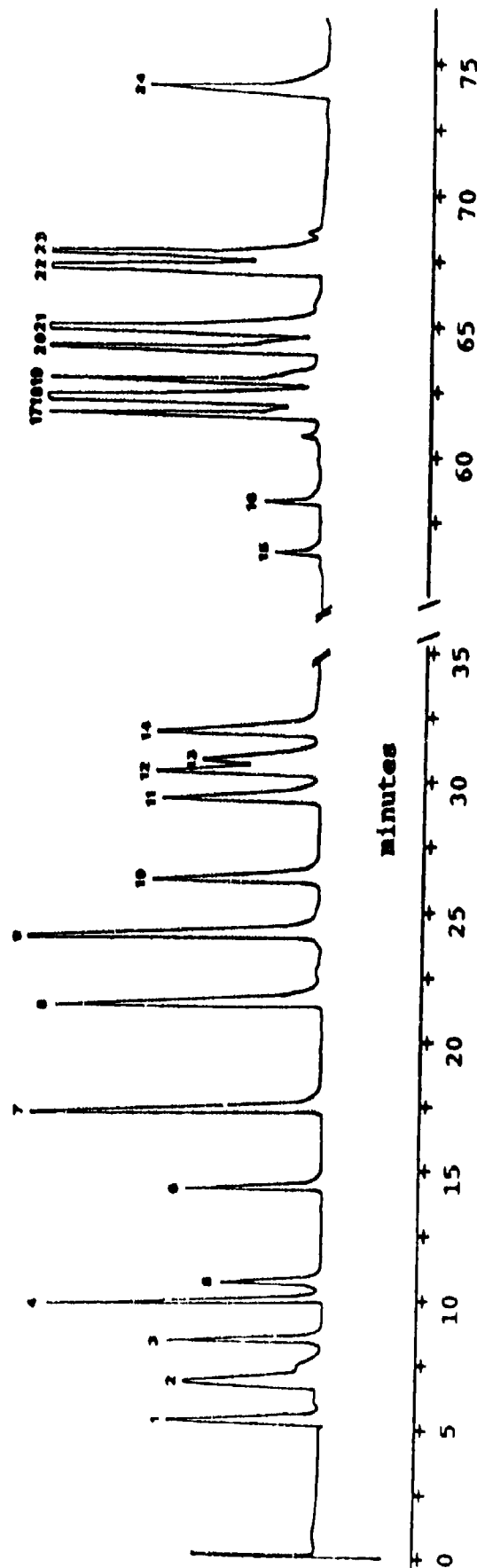
Figure 3.1 HPLC chromatogram showing the separation of a series of munition residues, environmental degradation products of explosives, and PAHs, using the gradient chromatographic (screening) method.

compounds that would otherwise be missed by previous methods, and produced sharp symmetrical elution peaks for all compounds tested. However this chromatography required 90 min to complete, and could not be used as a routine procedure at high sensitivity (compounds <1 mg L^{-1}) because of problems with baseline drift. The isocratic HPLC method of Miyares and Jenkins proved effective in quantitating intact RDX, TNT, and DNTs (2,4-, and 2,6-dinitrotoluene) in water, acetonitrile, and methanol but performed less well with the aminodinitrotoluenes because they were later eluting and exhibited significant peak broadening (Fig. 3.2). Peak broadening caused problems with quantitation because it caused erratic start times during electronic integration of peak areas. We also observed that this solvent and column combination was unusually sensitive to temperature. At room temperatures the large negative absorbance peak from acetonitrile interfered with the quantitation of HMX. At temperatures above 23°C retention times were shortened, and at 30°C the system no longer resolved the two aminodinitrotoluenes.

Recovery of explosives doped into uncontaminated soil were nearly quantitative (Table 3.2); adjustments of recoveries due to gain or loss of the DNB internal standard were insignificant. Conversely, recoveries from the soil and water after leaching experiments ranged from 10-15% for TNT, 2-5% for 2,4-DNT, and even less for 2,6-DNT. Due to these low recoveries of the nitroaromatics from the leached soils, the concentrations of explosives in soil extracts, and in aqueous leachates, were often diminished to levels below our criteria of detection. The criterion of detection is defined as the lowest certifiable limit for quantitation. The respective criteria of detection were calculated using the computerized Quality Assurance Program of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA),[28] based on the methods of Hubaux and Vos[29,30]. Criteria of detection values were determined separately for leachate (aqueous) and soil samples for each explosive and transformation product, with details and calculations given in Appendix B. Criteria of detection for selected compounds are presented in Table 3.3, as a function of sample matrix.

When a compound was identified but quantitated to be at levels below the criteria of detection, it was termed to be a "**trace**" quantity and identified as < **criterion of detection**; a **zero value (0)** was reported when "**no peak**" was registered by the integration unit of the HPLC (i.e. not detectable) under the analytical conditions described in this report (above).

27

1   Cyclotetramethylenetetranitramine (HMX)
2   1,3,5-Trinitrobenzene (TNB)
3   Cyclotrimethylenetrinitramine (RDX)
4   1,3-Dinitrobenzene (DNB)
5   2,4,6-Trinitrotoluene (TNT)
6   2,4-Dinitrotoluene (2,4-DNT)
7   2,6-Dinitrotoluene (2,6-DNT)
8   2-Amino-4,6-dinitrotoluene (2-Amino-DNT)
9   4-Amino-2,6-dinitrotoluene (4-Amino-DNT)
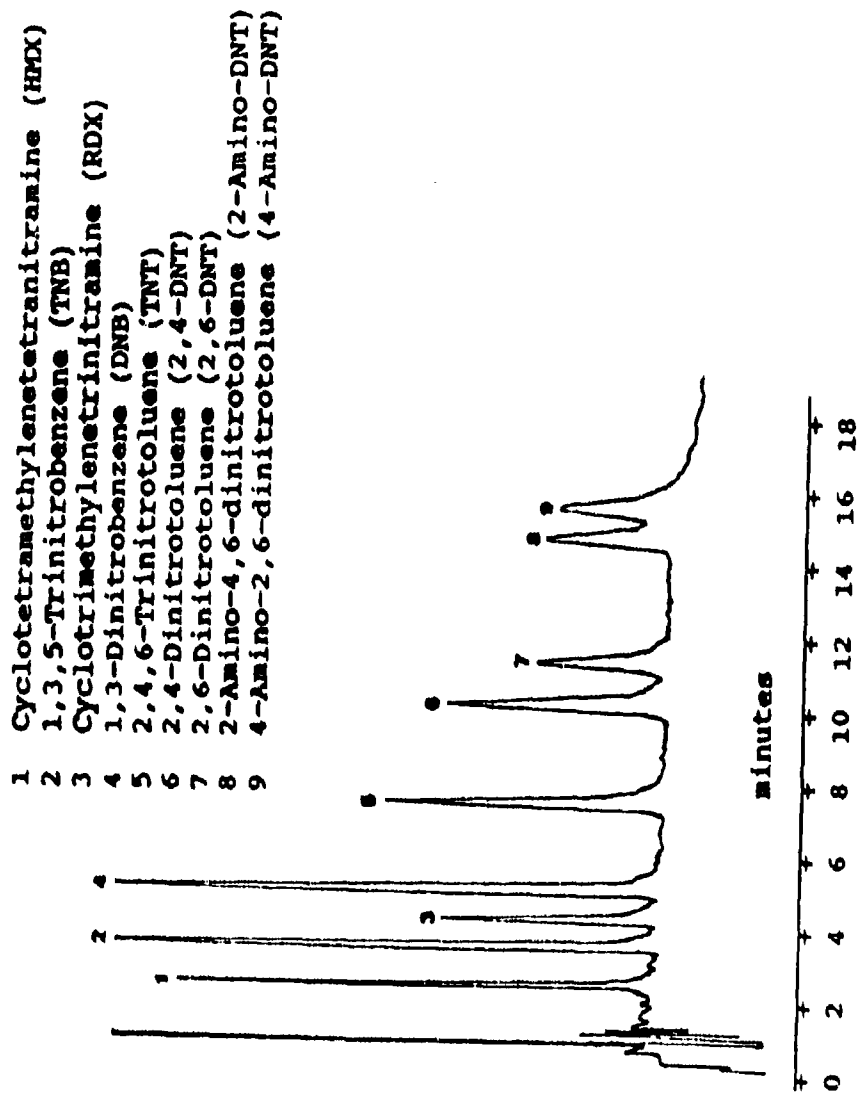
Figure 3.2 An example of the separation of a series of munition residues and associated co-contaminants, by the isocratic HPLC method12.

28

Table 3.3 Criteria of Detection* for Selected Explosives and Their Transformation Products for Leachate (Aqueous) and Soil Samples.

Compound	Criteria of Detection by Sample Matrix	
	Leachate $(mg\ L^{-1})$	Soil $(mg\ kg^{-1})$
RDX	0.07	5.8
HMX	0.14	2.9
TNT	0.09	6.1
2,4-DNT	0.17	5.7
2,6-DNT	0.37	5.2
2-Amino-DNT	0.14	15.4
4-Amino-DNT	0.12	14.6
TNB	0.15	2.4

* Calculations detailed in Appendix B.

c. Analytical Methods for Metals Determinations by Atomic Absorption Spectrophotometry

Concentrations of Cd, Cr, Cu, Pb, and Zn in uncontaminated soils and OB/OD contaminated ash/soil mixtures from each of the four OB/OD sites were determined in order to compare the background levels of metals in the respective soils with those of the contaminated/fortified (spiked) samples. Complete results from these analyses are reported in Appendix C. Duplicate 4.00 ±0.02 g air-dried subsamples from each of the uncontaminated, contaminated, and contaminated/fortified (spiked) soils were each heated for 3 h on a hot plate in 20 mL 1.0 M trace-metal grade HNO_3. When the samples were cool, each was filtered by gravity through Whatman #50 paper, then brought to 50-mL volume with ultrapure water (reverse osmosis followed by double-deionization). All samples were analyzed for total extractable Cd, Cr, Cu, Pb, and Zn levels by atomic absorption spectrophotometry (Perkin-Elmer Model 3030 AA Spectrometer).

Quality assurance and control (QA/QC) for the metal determinations were achieved as follows. Absorbance and

29

concentration values for standard solutions were initially assessed to assure compliance with the values listed in the Perkin-Elmer methods guide. Standard solutions of the metals were periodically reread (absorbance redetermined) throughout the analyses for each metal determined, to check for instrument drift. Blank solutions were analyzed to detect any possible metal contamination. Additional subsamples were selected at random and prepared in replicate, to verify the analytical results obtained in initial analyses.

4.

a. Results

i. Soil Parameters

The soil type at the RAAP OB/OD area consisted of Wheeling sandy loam soil (Fine-loamy, mixed, mesic, ultic, Hapludalfs),[18] thus soil of this type was sought in an uncontaminated area on-site. Physical and chemical analyses of soil from the uncontaminated site confirmed the Wheeling sandy loam soil type. These soil parameter results are presented in Table 4.1.

Table 4.1. Physical and Chemical Characteristics* of Wheeling Sandy Loam from the Uncontaminated RAAP Site.

	SURFACE A HORIZON (0-38 cm) 0-15 INCHES	SUB-SURFACE B HORIZON (38-61 cm) 15-24 INCHES
SAND %	70	70
SILT %	22	22
CLAY %	8	8
ORGANIC MATTER g/kg	20	17
CEC cmol$_c$/kg	8.2	9.7
pH	6.1	6.8

* Values represent replicate determinations by the University of Maryland Soil and Plant Testing Laboratory, College Park, MD.

Concentrations of all metals studied were higher in the contaminated than the uncontaminated Wheeling sandy loam soil (Appendix C). The concentration of each metal in contaminated soil was divided by the concentration in uncontaminated soil to reveal the anthropogenic elevation, in percent. Thus, relative concentrations of metals in contaminated soil were expressed as percentages of the values from uncontaminated background soil, followed by the determined concentration

values (mg kg^{-1}) for the contaminated soil: Cd 200% (1.1), Cr 160% (10.9), Cu 500% (30.0), Pb 1400% (183) and Zn 450% (303). On the basis of the anthropogenic elevations alone, the greatest potential environmental hazard from metallic residues at RAAP appears to be due to the elevated Pb concentrations in OB/OD contaminated soil.

Twelve uncontaminated Wheeling sandy loam soil columns having soil-core depths that were the most similar to the median were initially selected for preliminary evaluation in accordance with the procedures described in this report. Ten of these met the thru-put criteria while two did not. After replacing these two columns and testing the replacement columns, the set of twelve selected for spiking with contaminated RAAP soil and further investigation was successfully identified.

ii.   Leachates

The volumes of leachates collected are given as a function of time in Appendix D, Table D-1. Concentrations of munition residues in RAAP soil-core leachates were determined by HPLC methods described in Volume I, Sections 2.d, 3, and 4.d (above). There were no detectable concentrations of munition residues or transformation products in any of the leachates harvested from RAAP Wheeling sandy loam soils, whether from control soil-core columns or soils contaminated with explosives (Appendix D, Tables D-2 and D-3).

The average pH values for each leachate harvest are given in Table 4.2. Generally, the pH of leachates tended to be quite high, and slowly decreased as leaching of the soil-core columns progressed. However, the average pH of leachates differed by less than one pH unit over the course of 274 days, during which the soil-core columns received 2" (5 cm) per week of synthetic rain (pH 4.60 ±0.02). Since the pH of the native Wheeling sandy loam was 6.1 in the A horizon and increased to only 6.8 in the B horizon, the higher pH of the leachates from the treatment columns may in part be due to solubilization of munition ash materials, and the action of soil microorganisms, both in the soil and in the resulting leachates which become inoculated naturally.

iii.   Soil

Concentrations of munition residues in RAAP soils were determined by the HPLC methods described in this report. Results of analyses for each soil-core section, from all RAAP treatment and control soil-core columns, are given in Appendix D, Tables D-4.1 through D-4.12. The results from triplicate treatment soil-core columns are summarized in Table 4.3. The munition residues and transformation products that were

32

present in treatment soil-core columns at commencement of column leaching included TNT, TNB, 2,4-DNT, and 2,6-DNT. During this investigation of the transport and transformation of munition residues, the related compounds found in treatment soil-core columns included TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT.

Table 4.2 Average Leachate pH Values at Each Leachate Harvest Day for RAAP Soil-Core Columns that Received 1" (2.5 cm) Synthetic Rain (pH 4.60 ±0.02) Twice per Week for Up to 39 Weeks.

Days	12 columns	Average pH value	Standard Deviation
14		8.1	0.3
28		7.8	0.2
42		7.9	0.3
56		7.9	0.3
70		8.0	0.4
84		7.9	0.4
	8 columns		
98		7.9	0.4
112		7.8	0.4
127		7.8	0.4
144		7.8	0.3
155		7.8	0.3
168		7.6	0.3
183		7.6	0.4
	4 columns		
196		7.7	0.4
210		7.6	0.4
225		7.6	0.5
239		7.4	0.2
253		7.8	0.4
267		8.2	0.3
274		7.8	0.3

b. Discussion

        When the leaching of the soil-cores commenced, TNT, TNB, 2,4-DNT, and 2,6-DNT all were initially present in the top inch of soil of treatment columns (Table 4.3). TNT and TNB in the top inch of soil were extractable at 62 and 104 mg kg^{-1} (ppm) respectively. TNB, a transformation product of TNT that is exposed on the surface at OB/OD sites, is frequently found in surface soils at concentrations exceeding that of the parent compound,[31] and this was the case for the contaminated RAAP soil. When leaching commenced, no 2-amino-DNT or 4-amino-DNT (amino-DNTs) was present in the RAAP soil at detectable concentrations

33

even though in many environments TNT is microbially transformed by reduction to amino-DNTs.[32] The 2,4- and 2,6-DNTs were added uniformly in the top inch of RAAP soil at the concentrations of 125 mg kg^{-1} 2,4-DNT and 40 mg kg^{-1} 2,6-DNT, resulting in initial extractable concentrations in soil of 116 and 37 mg kg^{-1} respectively.

TNT, TNB, 2,4-DNT, and 2,6-DNT were transported vertically into the soil column, but to a depth of no more than three inches; as were 2-amino-DNT and 4-amino-DNT, unless created at depth within the RAAP treatment soils. After three months of leaching, the concentrations of extractable TNT, TNB, 2,4-DNT, and 2,6-DNT in the top inch (0-2.5 cm) of soil of treatment columns had declined by transformation of TNT *in situ* following transport. All of these munition residues migrated but to a very small degree, and resulted in very low concentrations only in the top 3" (0-7.5 cm) to 7, <1.3, <1.8, and <3.5 mg kg^{-1} respectively, and remained at these levels through six additional (nine total) months of leaching; while concentrations of these compounds in the second (2.5-5 cm) and third (5-7.5 cm) inches of soil were approximately the same or even lower. TNT and 2,6-DNT concentrations in the top inch of soil declined by a factor of ten (one order of magnitude) or more, while TNB and 2,4-DNT concentrations decreased by a factor of 100 (two orders of magnitude) or more. Only trace amounts of the amino-DNTs (primarily 2-amino-DNT) were found in the leached RAAP soil, and these amounts were formed *in situ* (since none was originally present). The amino-DNTs are substantially more polar compounds than their precursor TNT, and such increased polarity generally leads to increase water solubility and thus mobility. The amino group may also destabilize chemisorption, allowing amino-DNTs to undergo ion exchange reactions at negatively charged soil surfaces. Both of these characteristics would tend to favor migration of amino- DNTs. However, even though more polar than TNT, the amino-DNTs were also found only in the top three inches of the RAAP treatment soils and only in trace amounts.

The dramatic decline in the concentrations of the munition residues in the RAAP soil did not cause high concentrations of any of these compounds in RAAP leachates. Not even trace concentrations of any of the compounds (or any other transformation products) were found in any of the leachates from the contaminated RAAP soil cores. Thus in this Wheeling sandy loam soil, the rate that TNT was transformed to either form of amino-DNT occurred at a very slow rate, as evidenced by their low concentrations in the soil, lack of presence in leachates, and lack of additional transport over time.

TNT, TNB, 2,4-DNT, and 2,6-DNT all became bound within the A horizon of the RAAP soil, in response to the simulation of natural weathering processes (i.e. alternating wetting and drying cycles, with the surface of the soil exposed to sunlight). The time-dependent disappearance of these munition residues in the environment may very well be due to covalent or other non-equilibrium

Table 4.3 Average ±s Concentrations (mg kg^{-1}) of Acetonitrile Extractable TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT in 1" (2.5 cm) Sections of Triplicate Treatment Soil-Core Columns.

<u>TIME ZERO (NO LEACHING)</u>

DEPTH	TNT	TNB	2,4-DNT	2,6-DNT	2-amino-DNT	4-amino-DNT
				mg kg^{-1}		
1"	62	104	115.8	36.6	0	0
	±7	±1	±0.2	±0.8	-	-

Below this depth: No detectable concentrations (0).

<u>THREE MONTHS OF LEACHING</u>

DEPTH	TNT	TNB	2,4-DNT	2,6-DNT	2-amino-DNT	4-amino-DNT
0-1"	7	<1.3	<1.8	<3.5	<1.4	0
	±1	-	-	-	-	-
1-2"	2	<1.3	<1.8	0	<1.4	<1.0
	±1	-	-	-	-	-
2-3"	0	0	<1.8	0	<1.4	0
	-	-	-	-	-	-

Below this depth: No detectable concentrations (0).

<u>SIX MONTHS OF LEACHING</u>

DEPTH	TNT	TNB	2,4-DNT	2,6-DNT	2-amino-DNT	4-amino-DNT
0-1"	6	<1.3	<1.8	0	0	0
	±1	-	-	-	-	-
1-2"	<1.3	<1.3	<1.8	0	0	0
	-	-	-	-	-	-
2-3"	0	0	<1.8	0	0	0
	-	-	-	-	-	-

Below this depth: No detectable concentrations (0).

<u>NINE MONTHS OF LEACHING</u>

DEPTH	TNT	TNB	2,4-DNT	2,6-DNT	2-amino-DNT	4-amino-DNT
0-1"	6	<1.3	2.5	<3.5	<1.4	0
	±3	-	±1.6	-	-	-
1-2"	3	<1.3	<1.8	<3.5	<1.4	0
	±1	-	-	-	-	-
2-3"	0	0	0	0	<1.4	0
	-	-	-	-	-	-

Below this depth: No detectable concentrations (0).

bonding to natural soil components. The A horizon of mineral soils, such as that at RAAP, usually reflect the influence of accumulated organic matter. Organic matter in soil tends to bind TNT and related compounds quite strongly and in very substantial amounts[33] especially if the binding capacity of the soil for these compounds has not been exceeded. Furthermore, though the aromatic ring structure of these compounds is resistant to degradation, evidence exists of other additional environmental processes in which these compounds may also become strongly bound to soil.

The concentrations of these munitions in RAAP soils were determined by sonically extracting the soil with acetonitrile, an efficient extractant for TNT, 2,4-DNT and 2,6-DNT and related compounds. Thus any chemically related munition residues not extracted from soil by sonication with acetonitrile are so strongly bound that they are effectively unleachable.

5.   CONCLUSIONS

*   **Intact Soil Column System: CESMU**

    A state-of-the-art controlled environment soil-core microcosm unit
(CESMU) system was developed to determine the transport and transforma-
tion of chemicals in RAAP soil.   The system used intact soil-core
columns from the RAAP OB/OD site.   The soil cores were put into the
CESMU, where experiments were conducted under controlled environmental
conditions.   The major improvement of the CESMU system over existing
microcosm technology was incorporation of a controlled weak vacuum to
cause a continuous tension on the soil-core columns.   This tension
mir cked the effects of soil matric and gravitational tension in real
world systems, allowing study of chemical transport and transformation
under laboratory conditions.

*   **Explosives and Transformation Products in Leachates and Soil**

    There were no detectable concentrations of munition residues or
transformation products in any of the leachates harvested from RAAP
Wheeling sandy loam soils.

    During this investigation of the transport and transformation of
munition residues, the compounds found in treatment soil-core columns
included TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT.   All
of these were transported vertically into the soil column, but to a
depth of no more than three inches. TNT, TNB, 2,4-DNT, and 2,6-DNT all
became bound within the A horizon of the RAAP soil, in response to the
simulation of natural weathering processes (i.e. alternating wetting and
drying cycles, with the surface of the soil exposed to sunlight).

*   **Anthropogenic Elevation of Metal Levels in Soil**

    Concentrations of all metals studied were higher in the
contaminated than the uncontaminated Wheeling sandy loam soil. Relative
concentrations of metals in contaminated soil expressed as percentages
of the values from uncontaminated background soil, and determined
concentration values (mg kg^{-1}) for the contaminated soil, were: Cd 200%
(1.1), Cr 160% (10.9), Cu 500% (30.0), Zn 450% (303), and Pb 1400%
(183). On the basis of the anthropogenic elevations alone, the greatest
potential environmental hazard from metallic residues at RAAP appears to
be due to the elevated Pb concentrations in OB/OD contaminated soil.

37

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38

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APPENDIX A

QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

a. Analytical chemistry.

I. Analytical standards of explosives and related compounds
were prepared by purification of existing USABRDL standards.
Purification was accomplished by recrystallization in a water
acetone system. A mixture of HMX, TNB, RDX, TNT, 2,6DNT, 2,4DNT,
2-Amino DNT, and 4-Amino DNT was prepared from analytical
standards with each component at 100 ppm in acetonitrile. This
mixture was sealed and stored at 2 to 5 degrees centigrade and
used until expended (about six weeks).

II. The mixture was serially diluted with water or
acetonitrile in a ten step process to yield calibration standards
of 10, 5, 2.5, 1.25, 0.63, 0.32, 0.16, 0.08, 0.04, and 0.02 ppm.
The standards were analyzed, peak areas recorded and a plot of
concentrations/peak areas produced. Linear regression of this
data in the form of $Y = MX + B$ with concentration as the
dependent variable were calculated. This equation was used to
calculate unknown concentrations from analyzed peak areas. New
calibration standards were analyzed with each set of analytes run
and the calibration curve recalculated.

III. Control samples to be analyzed with the test samples
were prepared by diluting the multipart standard to 2.5 ppm with
acetonitrile. Control samples were prepared in triplicate and
analyzed with each batch of samples. The mean and standard
deviation of these analyses were calculated and results from each
analytical run plotted as scattergrams (Figures A1 to A9).

b. Extracts.

I. Soil columns were sectioned and soils ground and
extracted in accordance with SOP and all extracts analyzed in
triplicate. Quality assurance procedures were established to
ascertain the efficiency of the extraction process.
Uncontaminated soil samples were spiked after grinding with a
mixture of the compounds under study and a percent recovery
performed for each site (Table A1). Spiked samples were prepared
in triplicate and analyzed with each batch of 27 soil extracts.
II. Dinitrobenzene (DNB) was added to the acetonitrile soil
extraction solution as a means to provide an internal recovery
standard for each soil sample analyzed. Separate samples
containing only DNB and acetonitrile were analyzed in triplicate
with each batch of soil extracts. Mean recovery and standard
deviation of these samples were calculated as a check on
extraction losses and analytical imprecision. These results are
presented in Figure A10 .

43

c. Leachates.

Aqueous leachates were collected within the CESMU and removed for analysis. Samples were then refrigerated until analyzed. Leachates were not concentrated and recoveries were not corrected by internal standardization.

d. Measuring devices.

Soils and explosives were weighed on scales of certified accuracy. Pipets were checked for accuracy when placed in service. Volumetric glassware was of certified accuracy.

e. Quality Assurance Categories for Investigation.

This investigation was initiated prior to the Toxicology Division SOP MGT-1 of 1 Oct. 91. However, this work meets the criteria of "Exploratory Research" in nature and is therefore classified as a Category 1 investigation. Good Laboratory Practices as applicable to this category of investigation, which were in place at the onset of work (Jan 1989), were followed throughout.

Fig. A1
HMX VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.47   ◇ 2.78   △ 2.16

Appendix A

45
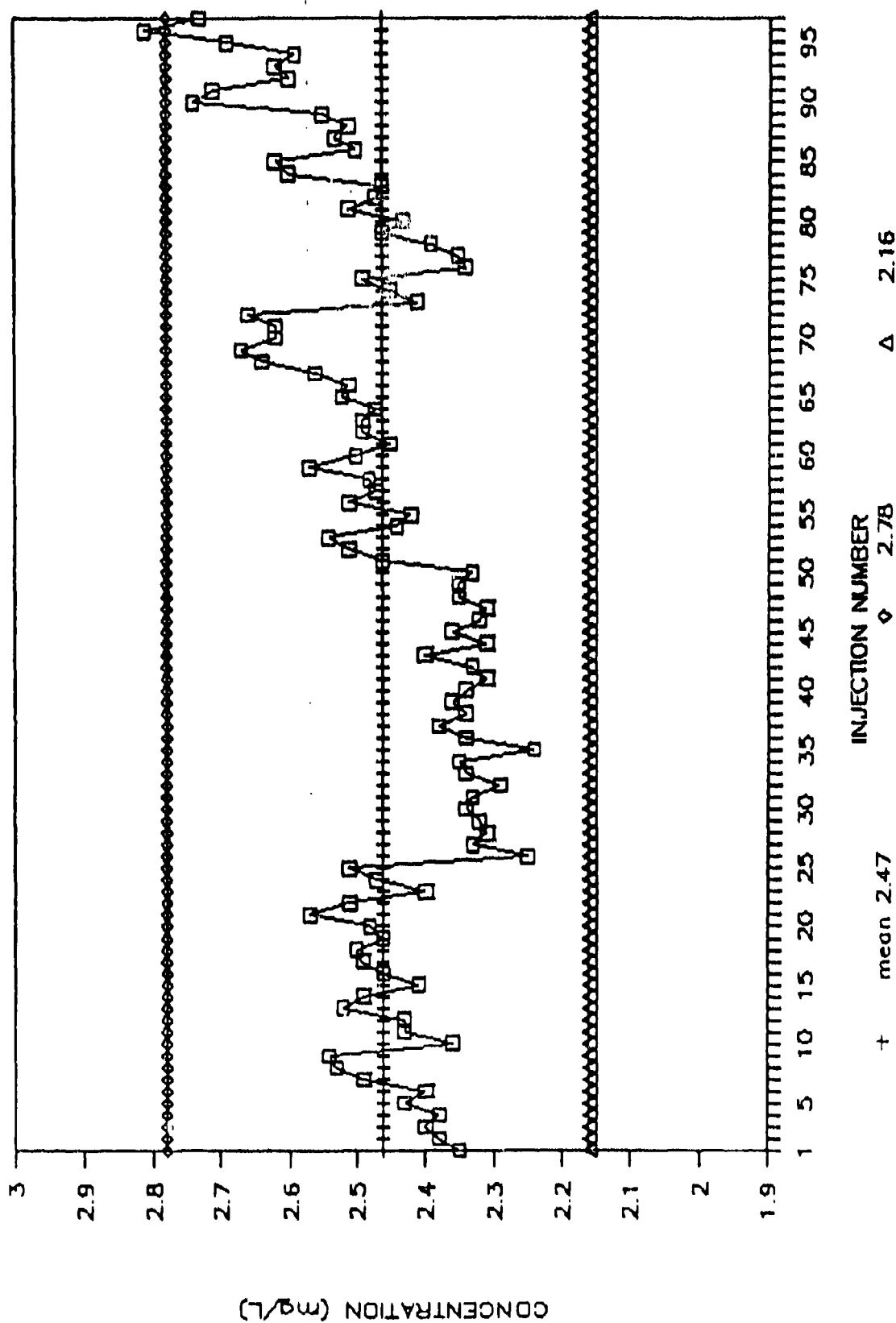
Fig. A2
TNB VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.54 ◇ 2.94 △ 2.14

Appendix A

46
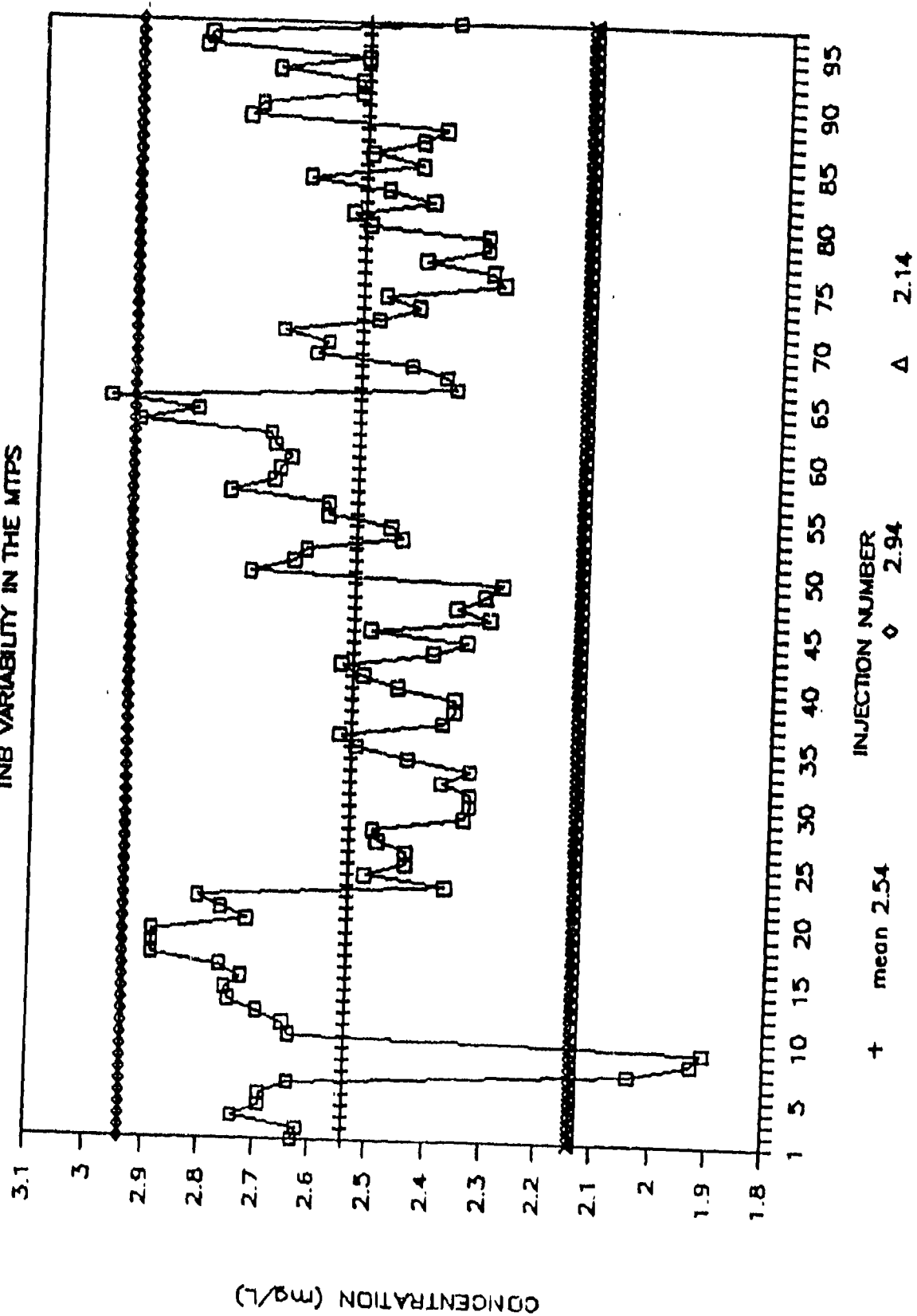
Fig. A3

RDX VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+   mean 2.55   ◇   3.06   △   2.10
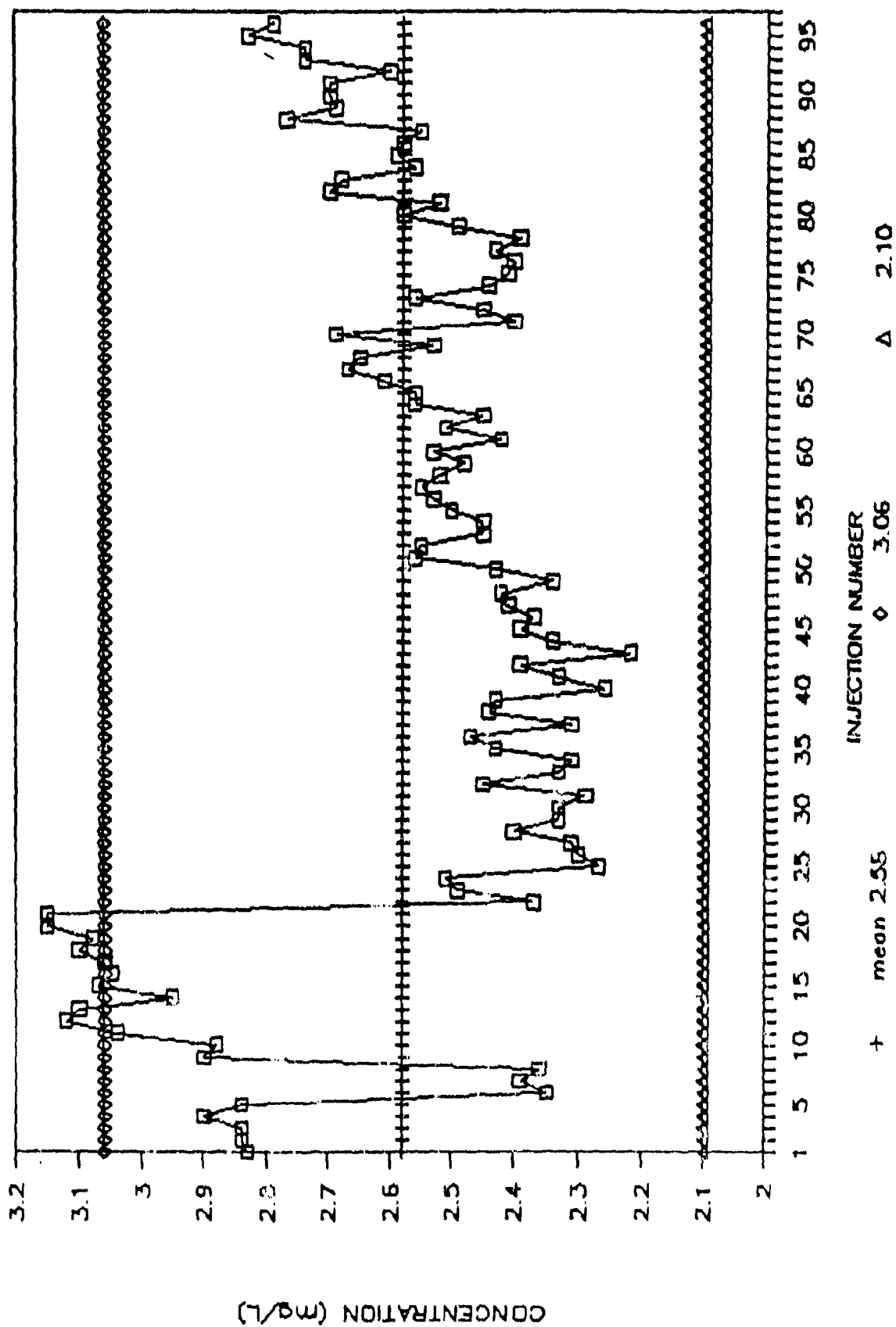
Fig. A4
TNT VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.59   ◇ 2.93   △ 2.25
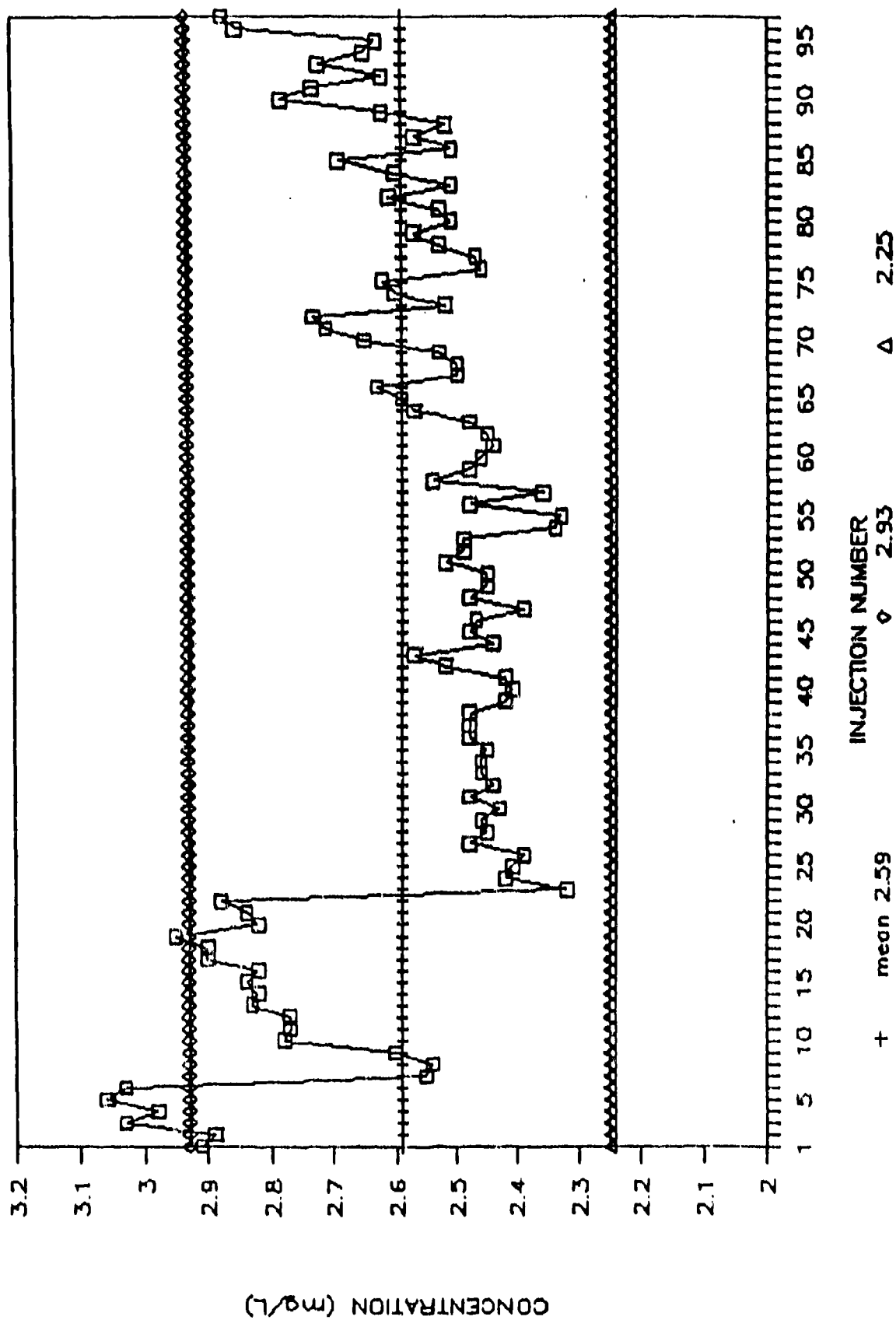
Fig. A5

2,4 DNT VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.66   ◇ 3.20   △ 2.12

Appendix A

49

Fig. A6

2,6 DNT VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.63   ◇ 3.20   △ 2.12
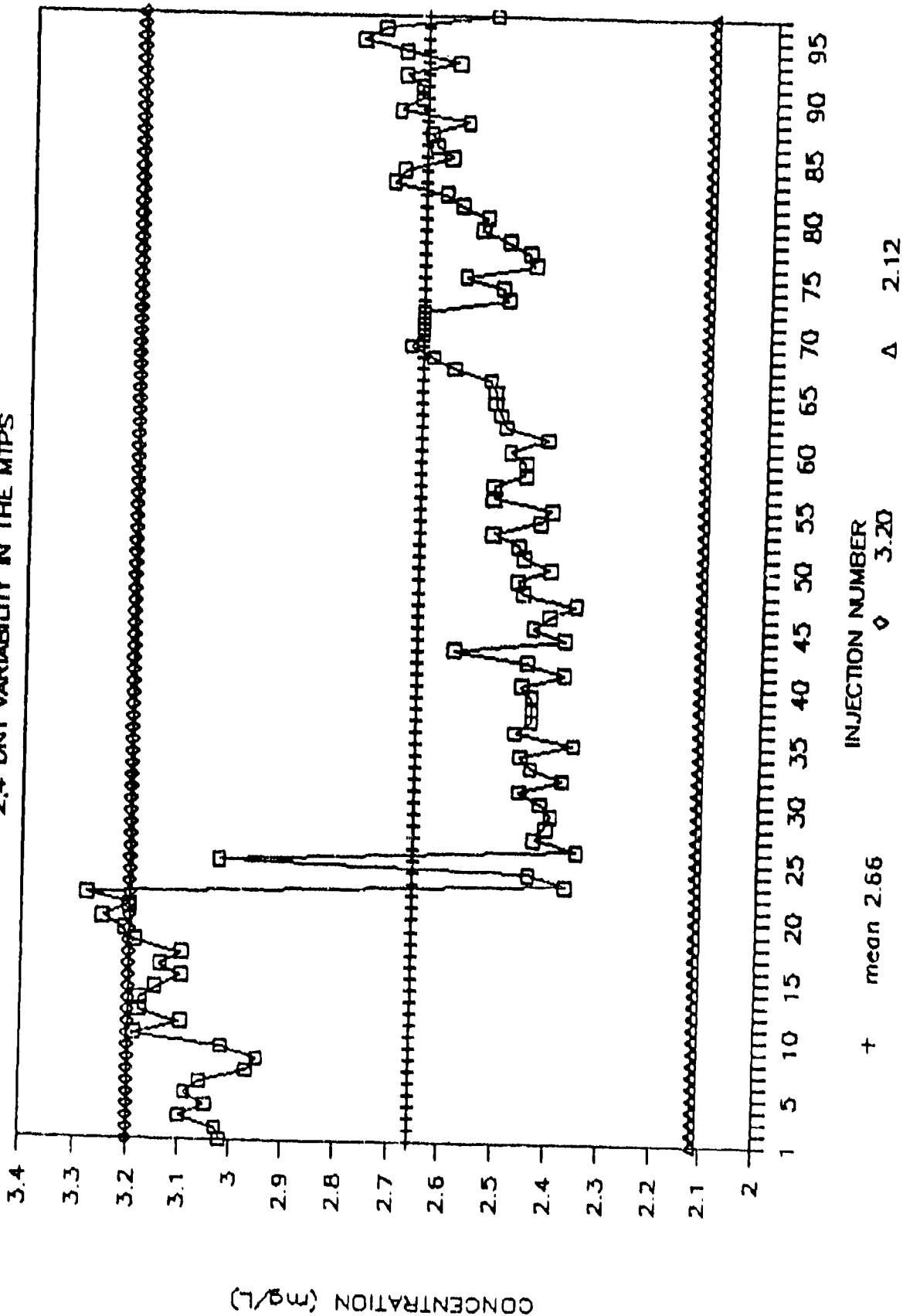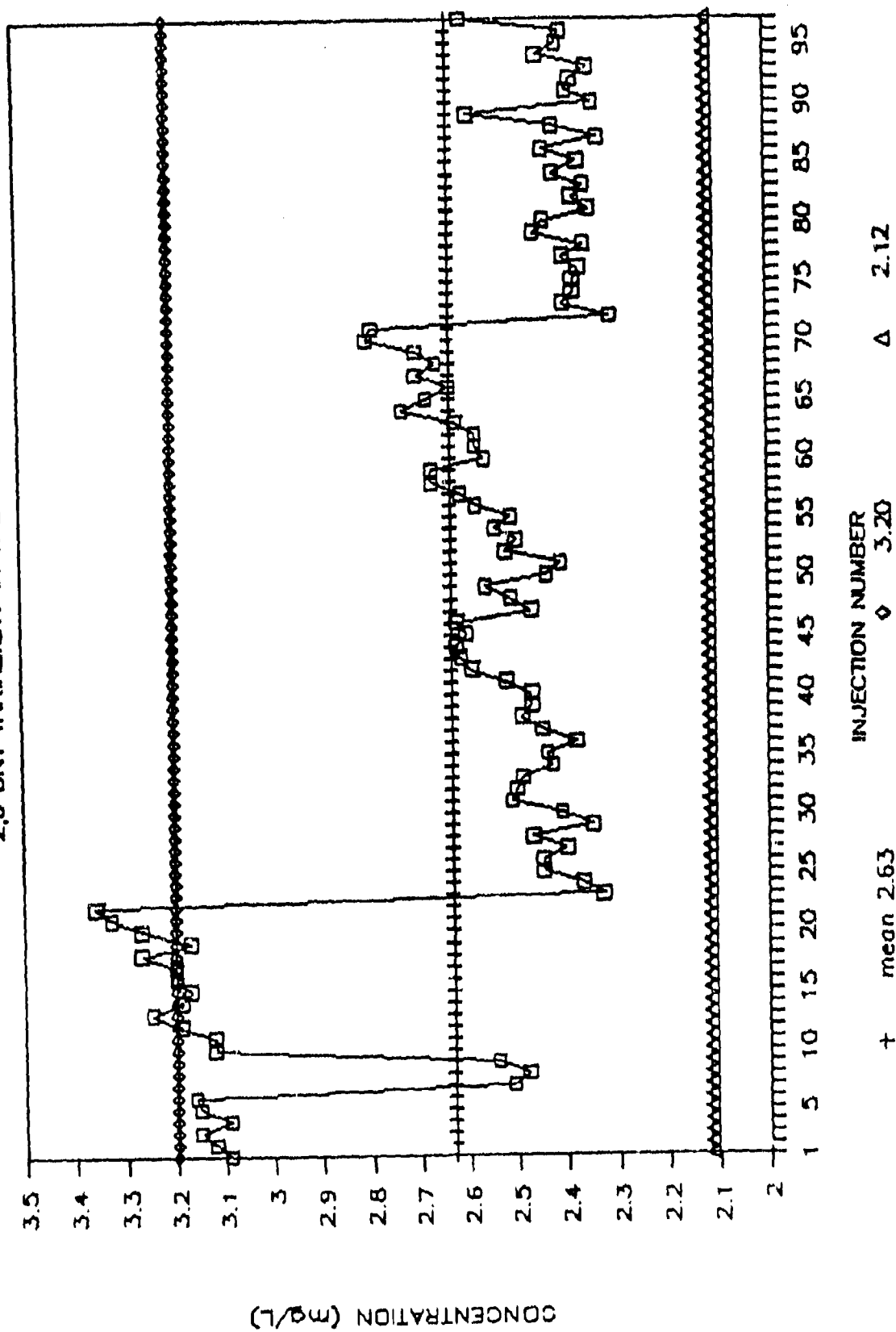
Fig. A7

2-AMINO 4,6 DNT VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.23   ◇ 2.43   △ 2.03
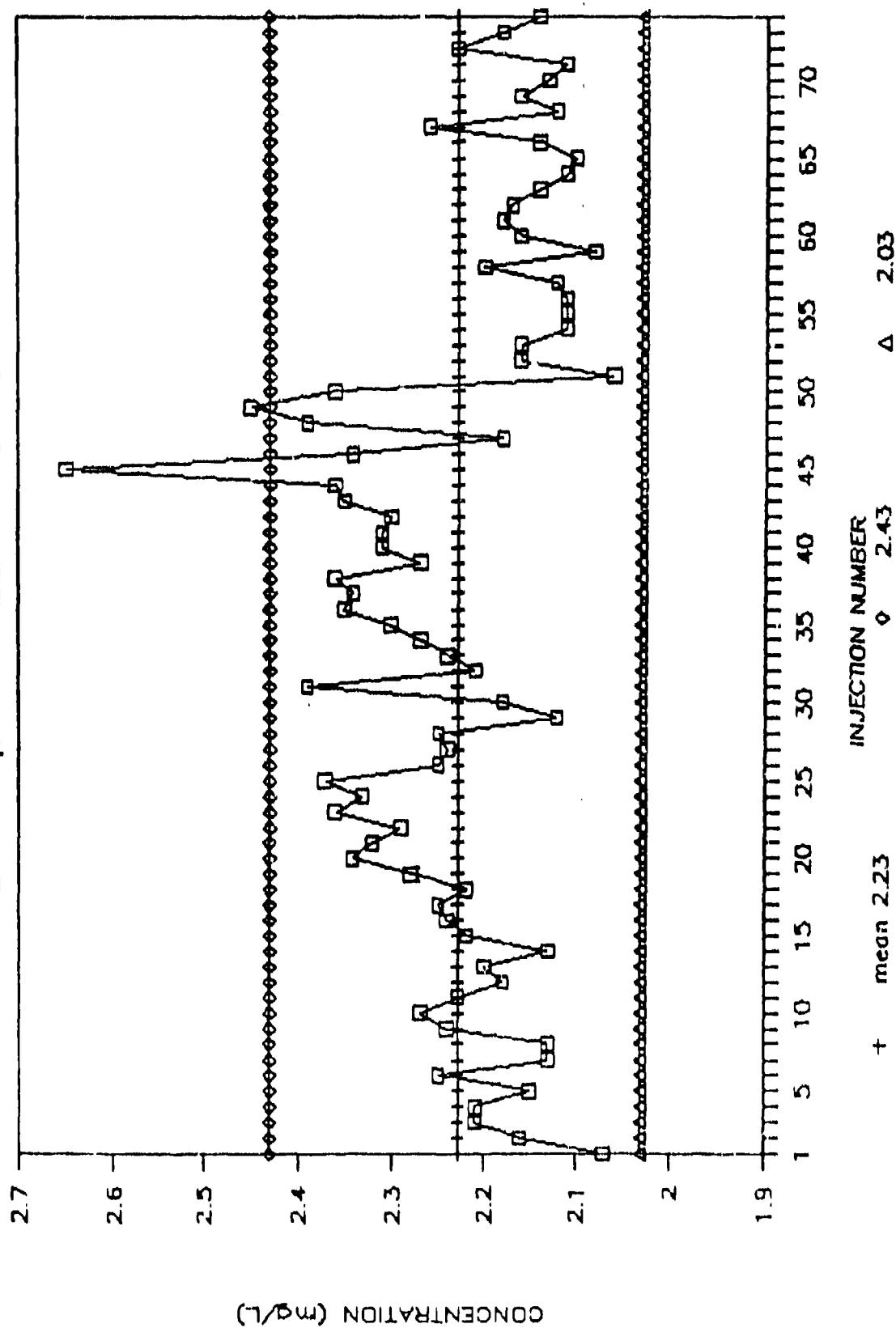
Fig. A8

4—AMINO 2,6 DNT VARIABILITY IN THE MTPS

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.17   ◇ 2.37   △ 1.97
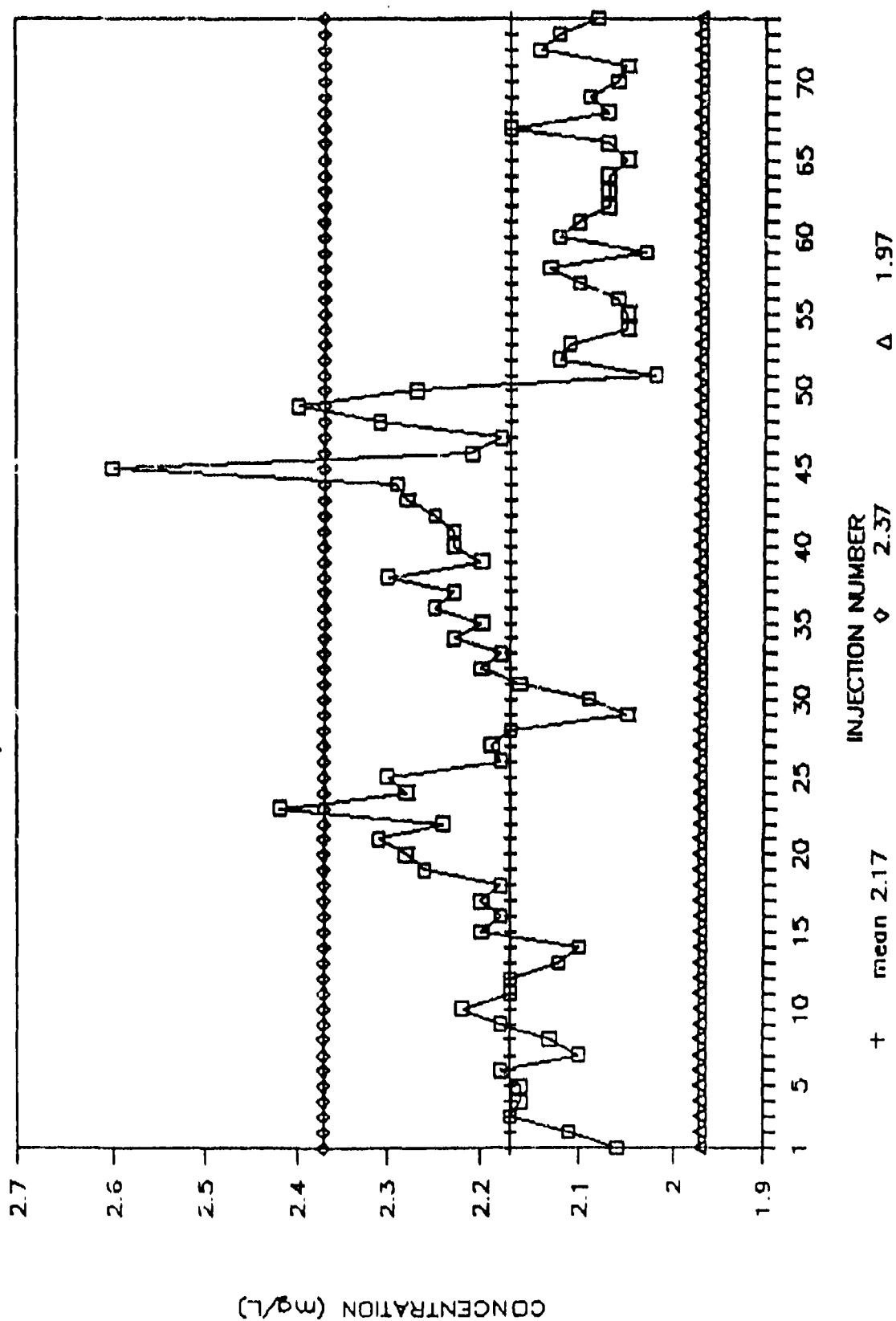
Fig. A9

DNB VARIABILITY IN THE MTPS

Appendix A

53
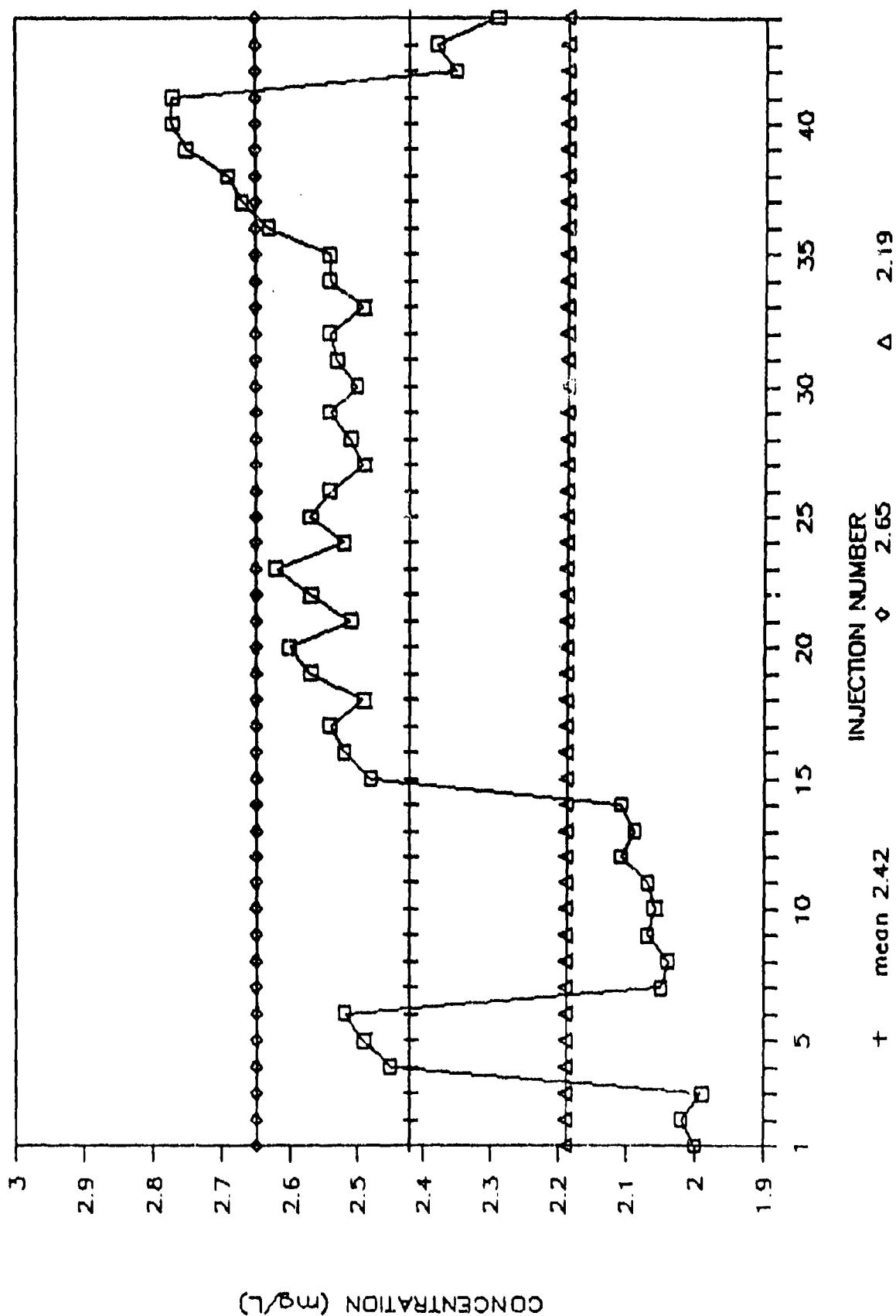
TABLE A1
PERCENT RECOVERY BY SITE

COMPOUND	RADFORD %RECOVERY	STD	MILAN %RECOVERY	STD
HMX	108.4	4.5	102.07	4.39
TNB	111.0	2.0	110.56	8.90
RDX	105.35	1.9	104.06	7.34
DNB	93.85	1.3	NONE	
TNT	99.50	1.2	108.91	6.74
2,4 DNT	103.45	1.3	107.24	6.84
2,6 DNT	100.95	1.9	107.02	8.81
2-AM 4,6 DNT	104.10	1.2	NONE	
4-AM 2,6 DNT	104.05	2.5	NONE	

COMPOUND	PUEBLO %RECOVERY	STD	ANNISTON %RECOVERY	STD
HMX	NONE		86.45	8.58
RDX	NONE		84.06	8.15
TNB	91.20	7.28	95.69	11.45
TNT	94.04	8.63	98.99	12.43
2,4 DNT	77.07	4.48	78.84	7.54
2,6 DNT	77.89	4.97	79.78	8.59
2-AM 4,6 DNT	57.63	14.43	73.48	21.87
4-AM 2,6 DNT	86.93	14.80	144.31	42.35

Fig. A10
DNB IN ACETONITRILE

CONCENTRATION (mg/L)

INJECTION NUMBER

+ mean 2.18   ◇ 2.25   △ 2.11

Appendix A

55
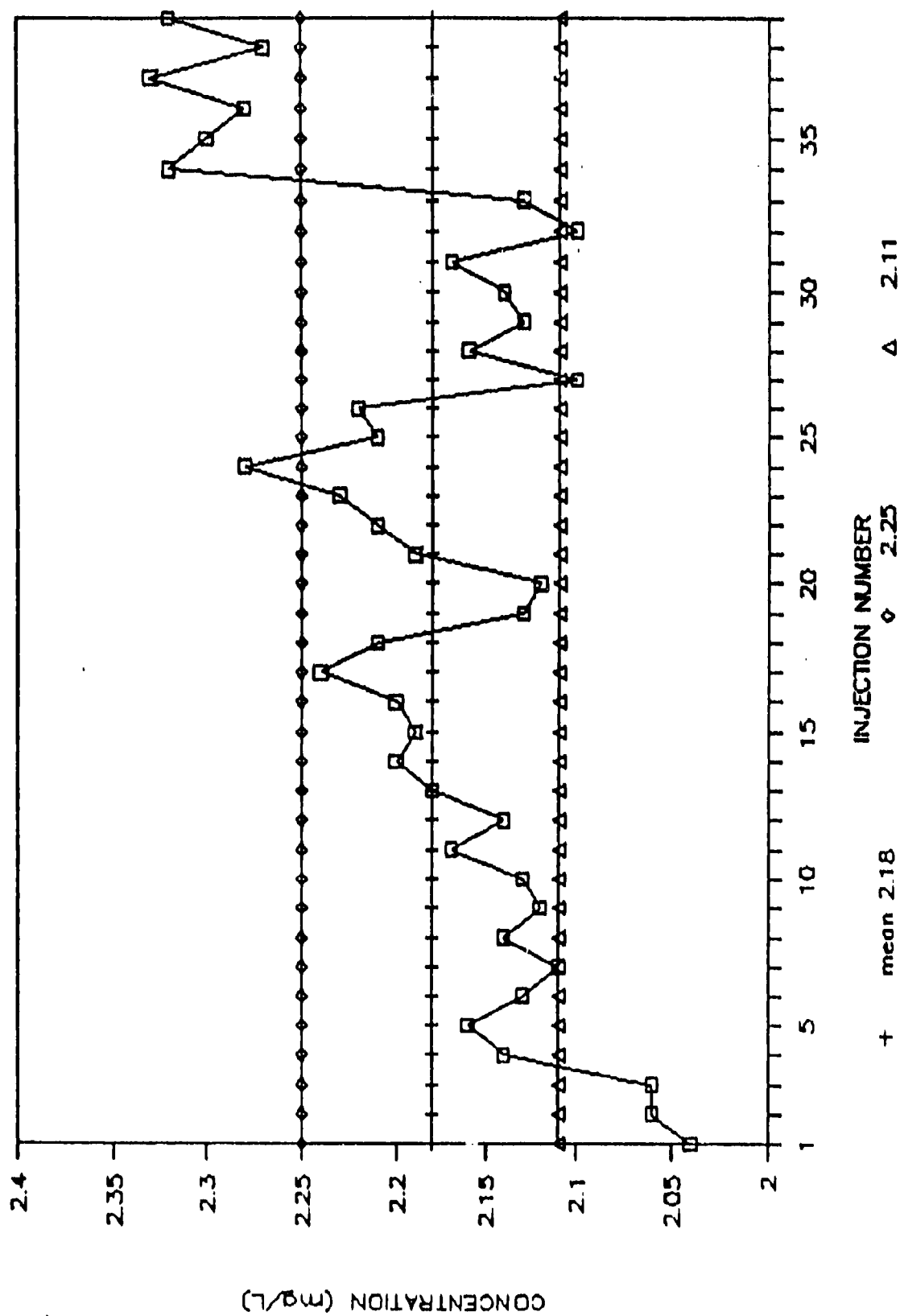
APPENDIX B

CRITERIA OF DETECTION

a. Explosives in Soil.

A criterion of detection (minimum accurate quantitation limit) was calculated from data of analysis of soil extracts in which the extraction and analysis steps were performed in triplicate and repeated in their entirety on four separate days. Criterion of detection of soil extracts was determined on a single soil type (Milan Soil). The soil was ground and sub-samples were spiked with 0.0, 0.4, 0.8, 1.63, 3.13, 6.25, 12.5, 25, and 50 mg/kg of a mixture of HMX, TNB, RDX, TNT, 2,4-DNT, 2,6-DNT, 2-AM, and 4-AM. For purposes of calculation the concentration of the explosives spiked onto the soil was assumed to be the "target concentration" in the soil at the time of analysis. The soils were extracted in the manner used for samples and the extracts analyzed. Target concentrations and the analytically derived values of the replicates were entered into the USATHAMA program for calculation of criteria of detection (Tables F1 - F8). This program generates a two dimensional plot with found values (analytically derived) as the dependent variable and target concentration as the independent variable (Figures F1 - F8). Linear regression of this relationship produces an equation in the form $Y = mx + b$ with;

Y = the found concentration
b = the found concentration intercept
m = the slope of the line

The variance about the regression line is plotted, thus generating parallel lines above and below the regression line. At the point where the line representing the mean minus the variance contacts the ordinate, values of Y can no longer be reliably distinguished from zero (Figures F9 - F16). Thus, criterion of detection is defined as the lowest concentration of analyte in an environmental sample which can be reliably distinguished from zero. Results of criterion of detection of soil extraction studies are summarized in Table F9. The criterion of detection levels from soil are:

Compound	Criterion of Detection
HMX	2.9 mg/kg
TNB	2.4 mg/kg
RDX	5.8 mg/kg
DNT	6.1 mg/kg
2,4-DNT	5.7 mg/kg
2,6-DNT	5.2 mg/kg
2-AM	15.4 mg/kg
4-AM	14.6 mg/kg

b. Explosives in Leachates.

In addition to the work done with soil extracts, criterion of detection was also performed for the leachates. The criterion of detection for these samples corresponds to the quantitation limit of the instrument because no sample preparation steps were employed.

The multipart standard containing HMX, TNB, RDX, TNT, 2,4-DNT, 2,6-DNT, 2-AM, and 4-AM was prepared at 1000 mg/L. This solution was diluted in a serial fashion to yield concentrations of 10, 5, 2.5, 1.25, 0.63, 0.32, 0.16, 0.08, 0.04, and 0.02 mg/L. These concentrations were analyzed in triplicate on four separate days and the results used to calculate the criterion of detection for each compound. Two separate criterion of detection studies were completed for the aqueous leachates and data from both studies are presented. Data from the first and second iteration of this work are identified by the small letter "a and b" after the table or figure number. For purposes of calculation the concentration of the explosives spiked into solution was the "target concentration". Target concentrations and the analytically derived values of the replicates were entered into the USATHAMA program for calculation of criteria of detection (Tables F10 - F17). This program generates a two dimensional plot with found values (analytically derived) as the dependent variable and target concentration as the independent variable (Figures F17 - F24). Linear regression of this relationship produces an equation in the form $Y = mx + b$ with;

Y = the found concentration
b = the found concentration intercept
m = the slope of the line

The variance about the regression line is plotted, thus generating parallel lines above and below the regression line. At the point where the line representing the mean minus the variance contacts the ordinate, values of Y can no longer be reliably distinguished from zero (Figures F25 - F32). Thus, criterion of detection is defined as the lowest concentration of analyte in an environmental sample which can be reliably distinguished from zero. Results of criterion of detection of leachate studies are summarized in Table F18. The criterion of detection levels for water and solvent are:

Compound	Criterion of Detection
HMX	0.14 mg/L
TNB	0.14 mg/L
RDX	0.12 mg/L
DNB	0.15 mg/L
TNT	0.09 mg/L
2,4 DNT	0.17 mg/L
2,6 DNT	0.36 mg/L
2-AM	0.14 mg/L
4-AM	0.14 mg/L

CERTIFICATION ANALYSIS   Report Date: 10/12/93
....

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: HMX   Analysis Date   03/18/92
                                               Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

```
        --- Model with Intercept ---      - Model through the Origin -
     Y = (-0.24876344) + (0.854201200)X       Y = (0.846765184)X
```

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	231.3894150	94	2.461589521	235.1184280	95	2.474930821
Total Error:	227.2558750	88	2.582453125	227.2558750	88	2.582453125
Lack of Fit:	4.133540000	6	0.688923333	7.862553000	7	1.123221857

```
        LOF F-Ratio(F): 0.266770896     LOF F-Ratio(F): 0.434943754
        Critical 95% F: 2.25            Critical 95% F: 2.17
```


ZERO INTERCEPT HYPOTHESIS
....
Zero Intercept Accepted   Calculated F: 1.514880108   Critical 95% F: 4
....

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

        Target Value Found Concentration

	Target Value	Found Concentration				
1:	50	41.500000	43.200000	42.300000	45.600000	46.500000
		48.500000	40.400000	41.900000	42.400000	39.700000
		38.900000	39			
2:	25	20.900000	21.400000	21.200000	22.900000	22.700000
		23	21.700000	21.700000	21.800000	19.400000
		19.400000	19.500000			
3:	12.500000	10.700000	10.600000	10.300000	9.9400000	9.2600000
		12.500000	10.400000	10.300000	9.6000000	10
		14.300000	1.2000000			
4:	6.2500000	5.2000000	4.5400000	4.8000000	5	5.0900000
		5.1900000	5.1000000	4.8000000	5.1000000	5.1900000
		4.9000000	4.9000000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: HMX   Analysis Date   03/18/92
                                                 Matrix: SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

          Target Value   Found Concentration

5:      3.1300000   2.6700000   2.4800000   2.4800000   2.7000000   2
                       2.3000000   2.7700000   2.6700000   2.4800000   2.5000000
                       2.5000000   2.6000000
6:      1.5600000   1.1200000   1.9000000   1.2100000   1.0300000   1.2200000
                       1.8000000   1.3200000   0.9300000   0.6400000   1.4000000
                       1.1000000   0.9900000
7:      0.8000000   0.8400000   0.7000000   0.6500000   0.6400000   0.7300000
                       0.5400000   0.4400000   0.5400000   0.5400000   0.6400000
                       0.2500000   0
8:      0.4000000   0.4400000   0.6900000   0.6100000   0   0
                       0   0   0   0   0
                       0   0

       *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93
....

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: TNB   Analysis Date   03/18/92
                                            Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

        --- Model with Intercept ---   - Model through the Origin -
     $Y = (0.141512116) + (0.905973870)X$ $Y = (0.910203938)X$

              (SS)   (df)   (MS)   (SS)   (df)   (MS)
Residual: 176.8768300 94   1.881668404 178.0835540 95   1.874563726
Total Error: 168.7549830 88   1.917670261 168.7549830 88   1.917670261
Lack of Fit: 8.121847000   6   1.353641167 9.328571000   7   1.332653000

     LOF F-Ratio(F): 0.705877957   LOF F-Ratio(F): 0.694933340
     Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

....
Zero Intercept Accepted   Calculated F: 0.641305342   Critical 95% F: 4
....

                    ****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

        Target Value Found Concentration

1:    50   45.600000 47.500000 46.100000 43.300000 43.800000
                  51.600000 42   45.300000 46.100000 45.900000
                  44.900000 45.400000
2:    25   23   22.900000 22.900000 23.400000 23.500000
                  23.500000 18.900000 21.300000 20.400000 23.900000
                  23.700000 23.800000
3:    12.500000   11.900000 11.700000 11.300000 10.900000 7.4700000
                  5.6300000 12.900000 11.700000 11.200000 11.600000
                  12   12.700000
4:    6.2500000   5.9100000 5.9100000 6.0900000 5.7000000 5.3000000
                  5.6800000 5.9100000 5.8600000 5.8000000 7
                  7.2000000 6.8000000

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: TNB   Analysis Date   03/18/92
                                             Matrix: SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

        Target Value   Found Concentration

5:      3.1300000   4.2000000   4.2000000   4.1000000   3.0400000   3.0400000
                       2.7500000   3.1000000   2.2900000   2.2300000   2.8600000
                       2.9800000   2.9200000
6:      1.5600000   1.4800000   1.4800000   1.5400000   1.5400000   0.8500000
                       1.0800000   1.2000000   1.5400000   2.8000000   1.3700000
                       2.3000000   2.9000000
7:      0.8000000   0.2300000   0.2200000   0.2100000   0.6200000   0.6200000
                       0.5600000   0.7900000   0.5100000   0.3300000   0.9100000
                       0.9100000   0.7900000
8:      0.4000000   0.2900000   2.6000000   2.6000000   2   2
                       0   0   0   0   0
                       0   0

        ***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

Appendix B   61

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

Method Name:	SOIL EXTRACTION	Units of Measure:	mg/Kg
Method Number:	1	Laboratory:	RW
Compound:	RDX	Analysis Date	03/18/92
		Matrix:	SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   · Model through the Origin ·
$Y = (-0.11490761) + (0.744807248)X$ $Y = (0.741372440)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	703.3546070	94	7.482495819	704.1502500	95	7.412107895
Total Error:	684.0883830	88	7.773731625	684.0883830	88	7.773731625
Lack of Fit:	19.26622400	6	3.211037333	20.06186700	7	2.865981000

LOF F-Ratio(F): 0.413062540   LOF F-Ratio(F): 0.368675063
Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS
- -
Zero Intercept Accepted   Calculated F: 0.106333905 Critical 95% F: 4
- -

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

        Target Value Found Concentration

1:	50	38.800000	39.900000	38.300000	25.900000	26.400000
		42.100000	39.700000	40.200000	40.020000	39.500000
		38.700000	38.700000			
2:	25	19.500000	19.800000	20.400000	19.500000	19.500000
		19.100000	6.2100000	12	11.500000	21.400000
		21.400000	21.100000			
3:	12.500000	10	10.100000	9.2500000	9.4000000	9.1000000
		2.4200000	11.700000	10.500000	10.100000	15.100000
		10.800000	10.800000			
4:	6.2500000	5.5000000	6	4.8000000	5	5.1500000
		4.8500000	4.6000000	4.6000000	4.2400000	4.4000000
		5.1500000	4.8500000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

Method Name:	SOIL EXTRACTION	Units of Measure:	mg/Kg
Method Number:	1	Laboratory:	RW
Compound:	RDX	Analysis Date	03/18/92
		Matrix:	SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value	Found Concentration				
5:	3.1300000	2.2700000	2.1200000	2.1200000	2.4000000	0.6100000
		0.7600000	2.1200000	2.2700000	2.4300000	2.2000000
		2.3000000	2.8000000			
6:	1.5600000	2	1.7000000	1.2000000	0.4500000	1.0600000
		1.0600000	0.4500000	0	0	0.6100000
		1.6700000	1.0600000			
7:	0.8000000	0	0	0	0	0
		0	0	0	1.3000000	1
		1.7000000	0.9200000			
8:	0.4000000	0.9000000	0	0	0	0
		0	0	0	0	0
		0	0			

***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: TNT   Analysis Date   03/19/92
                                               Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
$Y = (-0.03971536) + (0.884832944)X$ $Y = (0.883644807)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	1095.426110	94	11.65346926	1095.521060	95	11.53180063
Total Error:	1069.960770	88	12.15864511	1069.960770	88	12.15864511
Lack of Fit:	25.46534000	6	4.244223333	25.56029000	7	3.651470000

LOF F-Ratio(F): 0.349070418   LOF F-Ratio(F): 0.300318824
Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS
- -
Zero Intercept Accepted   Calculated F: 0.008147788   Critical 95% F: 4
- -

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

      Target Value   Found Concentration

1:	50	50.600000	46.800000	51.200000	28.300000	27.200000
		56.700000	45.700000	47.700000	47.700000	40.200000
		41.300000	41.400000			
2:	25	20.700000	19.700000	20.600000	22.400000	23.700000
		23.100000	14.800000	25.500000	26.300000	24.600000
		23.800000	25.300000			
3:	12.500000	12.600000	10.800000	10.500000	13.200000	6.4400000
		10.400000	11.300000	12.300000	11.600000	14.100000
		13.700000	17.800000			
4:	6.2500000	8.3000000	3.7000000	7.7000000	5.7000000	5.6300000
		5.9200000	4.2000000	5	5.3400000	5.9200000
		5.5600000	5.5600000			

Table F4 (Cont.)

CERTIFICATION ANALYSIS   Report Date: 10/12/93
....

Method Name:	SOIL EXTRACTION		Units of Measure: mg/Kg
Method Number:	1		Laboratory: RW
Compound:	TNT		Analysis Date 03/19/92
			Matrix: SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value	Found Concentration				
5:	3.1300000	2.1800000	2.7800000	2.7800000	2.2000000	1.5300000
		1.1600000	2.4800000	1.9700000	2.2600000	2
		2.1000000	1.8000000			
6:	1.6500000	1.2000000	1.4000000	1.5000000	1.8200000	1.5300000
		0.9400000	1.2400000	1.4600000	1.6000000	1.3100000
		0.9400000	0.9500000			
7:	0.8000000	0.2600000	0.6500000	0.5800000	0.5800000	0
		0	0	0	0	1
		0.7000000	0			
8:	0.4000000	0	0	0	0	0
		0	1.6500000	0	0	0
		0	0			

***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93

. . . .

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: 2,4DNT   Analysis Date   03/19/92
                                                Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

```
          ··· Model with Intercept ···    - Model through the Origin -
      Y = (-0.59402705) + (0.809804126)X     Y = (0.792047521)X
```

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	792.6388120	94	8.432327787	813.9022350	95	8.567391947
Total Error:	777.3167500	88	8.833144886	777.3167500	88	8.833144886
Lack of Fit:	15.32206200	6	2.553677000	36.58548500	7	5.226497857

```
      LOF F-Ratio(F): 0.289101677    LOF F-Ratio(F): 0.59169162ℓ
      Critical 95% F: 2.25           Critical 95% F: 2.17
```


ZERO INTERCEPT HYPOTHESIS

. . . .
Zero Intercept Accepted   Calculated F: 2.521655175   Critical 95% F: 4
. . . .

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

        Target Value Found Concentration

	Target Value					
1:	50	41.400000	40.200000	41.300000	40.200000	42.900000
		41.500000	26.700000	26.900000	43.200000	42.500000
		45.700000	46.400000			
2:	25	20.200000	21.200000	20.400000	12.500000	10.500000
		13.600000	23.700000	23.700000	23.900000	22.600000
		20.600000	19.700000			
3:	12.500000	12.100000	10.300000	16	10.200000	9.6200000
		9.4700000	9.4200000	6.4000000	6.7100000	11.400000
		11.800000	10.900000			
4:	6.2500000	4.5600000	4.8700000	5.3300000	2.9000000	5
		2.9500000	4.7100000	3.1800000	4.2500000	2.8000000
		3.4000000	3.2000000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

		Units of Measure:	mg/Kg
Method Name:	SOIL EXTRACTION	Laboratory:	RW
Method Number:	1	Analysis Date	03/19/92
Compound:	2,4DNT	Matrix:	SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value Found Concentration

	Target Value	Found Concentration				
5:	3.1300000	2	2.1000000	1.8000000	2.7200000	1.7200000
		2.4900000	1.9000000	1.0300000	1.0300000	1.2600000
		1.6400000	0.0300000			
6:	1.5600000	0.5700000	0.5700000	0.5700000	0	0
		0	0.2600000	0.4900000	0	1.4000000
		1.4000000	1.5000000			
7:	0.8000000	0	0	0.8000000	0	0
		0	0	0	0	0
		0	0			
8:	0.4000000	0	0	0	0	0
		0	0.9900000	0	0	0
		0	0			

***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93

....

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: 2,6DNT   Analysis Date   03/19/92
                                              Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   · Model through the Origin ·
$Y = (-0.58428181) + (0.824346024)X$ $Y = (0.806880723)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	681.4978330	94	7.249976947	702.0693100	95	7.390203263
Total Error:	643.8581280	88	7.316569636	643.8581280	88	7.316569636
Lack of Fit:	37.63970500	6	6.273284167	58.21118200	7	8.315883143

LOF F-Ratio(F): 0.857407840   LOF F-Ratio(F): 1.136582245
Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

....
Zero Intercept Accepted   Calculated F: 2.837454126   Critical 95% F: 4
....

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value Found Concentration

	Target Value	Found Concentration				
1:	50	39.900000	31.400000	39.300000	44.300000	45.600000
		47.200000	42.843000	44.500000	39.500000	42.200000
		40.600000	24.600000			
2:	25	21.400000	21	20.400000	25.200000	24.100000
		24.200000	23.300000	22.400000	20.200000	20
		19.900000	14.500000			
3:	12.500000	10.900000	9.6200000	10.500000	9.8600000	6.6600000
		5.2400000	4.2900000	10.300000	11.700000	9.8600000
		11.100000	11.500000			
4:	6.2500000	2.8000000	3.4000000	3.2000000	4.4100000	5.1200000
		5.2400000	3.3000000	3.8000000	2.2700000	4.4100000
		4.7200000	4.5300000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
....

		Units of Measure: mg/Kg
Method Name:	SOIL EXTRACTION	
Method Number:	1	Laboratory: RW
Compound:	2,6DNT	Analysis Date 03/19/92
		Matrix: SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value					
5:	3.1300000	3.1000000	1.2000000	2.6300000	1.3000000	0.3700000
		0.4900000	1.0800000	1.3200000	1.4400000	3
		2.9000000	0			
6:	1.5600000	1.9000000	2.2000000	1.7000000	0	0
		0	0	0.2500000	0.6100000	0
		0	0			
7:	0.8000000	1.2000000	0	2	1.8000000	0
		0	0	0	0	0
		0	0			
8:	0.4000000	0	0	0	1.8000000	0
		0	0	0	0	0
		0	0			

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: 2-AM   Analysis Date   03/19/92
                                             Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
$Y = (-0.73266610) + (0.786218675)X$ $Y = (0.764317883)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	5512.399130	94	58.64254394	5544.746050	95	58.36574789
Total Error:	5418.396520	88	61.57268773	5418.396520	88	61.57268773
Lack of Fit:	94.00261000	6	15.66710167	126.3495300	7	18.04993286

LOF F-Ratio(F): 0.254448884   LOF F-Ratio(F): 0.293148367
Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS
. . . .
Zero Intercept Accepted   Calculated F: 0.551594761   Critical 95% F: 4
. . . .

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value					
1:	50	60.400000	66.600000	70.400000	21	21.100000
		21.700000	35	47.500000	49.300000	17.400000
		18	24.100000			
2:	25	21.500000	15.300000	31.300000	38	33.400000
		13.500000	12.800000	12.500000	15.300000	16.400000
		18.300000	26.600000			
3:	12.500000	10.900000	10.500000	9.8600000	6.6600000	5.2400000
		4.2900000	10.300000	11.700000	9.8600000	7.6000000
		6.1000000	7.1000000			
4:	6.2500000	2.8000000	3.5000000	3.2000000	6.3500000	4.9400000
		1.2400000	4.7000000	4.3000000	2.3000000	3.8800000
		3.1800000	4.4100000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
- -

Method Name:	SOIL EXTRACTION	Units of Measure:	mg/Kg
Method Number:	1	Laboratory:	RW
Compound:	2-AM	Analysis Date	03/19/92
		Matrix:	SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value	Found Concentration				
5:	3.1300000	1.0600000	2.1200000	0.1800000	0.9000000	0.5400000
		0.3600000	1.0700000	1.7700000	1.6000000	1.6000000
		1.3000000	0			
6:	1.5600000	0.4000000	0.9000000	0.1000000	0.7000000	0
		0	0	0	0	0
		0	0			
7:	0.8000000	0	0	0	0	0
		0	0	0.3000000	0.1000000	0.6000000
		0	0			
8:	0.4000000	0	0	1.8200000	0	0
		0	0	0	0	0
		0	0			

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93

. . . .

Method Name:	SOIL EXTRACTION	Units of Measure:	mg/Kg
Method Number:	1	Laboratory:	RW
Compound:	4-AM	Analysis Date	03/19/92
		Matrix:	SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
Y = (-0.63682244) + (0.745388360)X   Y = (0.726352519)X

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	4427.118830	94	47.09700883	4451.556370	95	46.85848811
Total Error:	4191.612510	88	47.63196034	4191.612510	88	47.63196034
Lack of Fit:	235.5063200	6	39.25105333	259.9438600	7	37.13483714

LOF F-Ratio(F): 0.824048665   LOF F-Ratio(F): 0.779620173
Critical 95% F: 2.25   Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

. . . .
Zero Intercept Accepted   Calculated F: 0.518876689 Critical 95% F: 4
. . . .

****

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

Target Value   Found Concentration

	Target Value					
1:	50	23	22.400000	25.600000	21.300000	37.600000
		35.600000	32.261000	6.4400000	67.600000	47.700000
		51.600000	47.700000			
2:	25	12.500000	13.100000	12.800000	12.800000	21.300000
		14.500000	19.800000	37.600000	35.600000	32.500000
		28.700000	16.100000			
3:	12.500000	9.9000000	8.3000000	7.7700000	9.1900000	6.5200000
		7.7700000	12.700000	15.800000	14.700000	7.1000000
		8.2000000	8.1000000			
4:	6.2500000	3.4000000	2.5000000	3.4000000	2.9700000	2.6200000
		2.7900000	3.1500000	2.9000000	1.9000000	3.1500000
		2.9700000	4.2100000			

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: SOIL EXTRACTION   Units of Measure: mg/Kg
Method Number: 1   Laboratory: RW
Compound: 4-AM   Analysis Date   03/19/92
                                                 Matrix: SF

TABLE OF DATA POINTS   Targets: 8   Measures per Target: 12

          Target Value   Found Concentration

5:	3.1300000	0.1300000	0	0.1300000	0	0
		0	0	0	0	0.7000000
		0.7000000	0.3400000			
6:	1.5600000	0	0	0	0	0
		0	0	0	0	0
		0	0			
7:	0.8000000	0	0	0	0	0
		0	0	0	0	0
		0	0			
8:	0.4000000	0	0	0	0	0
		0	0	0	0	0
		0	0			

          *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Appendix B   73

Figure F1

HMX

RESPONSE UG/L

TARGET CONC UG/L

Figure F2

TNB

Figure F3

RDX

Figure F4

TNT

RESPONSE UGL

TARGET CONC
UGL

Figure F5

2, 4DNT

RESPONSE UG/L

TARGET CONC UG/L

Figure F6

2,6DNT

TARGET CONC
UGL

Figure F7

2-AM

TARGET CONC
UGL

RESPONSE

UGL

Figure F8

4-AM

Figure F9

HMX

TARGET CONC
UGL

FOUND CONC
UGL

Figure F10

TNB

TARGET CONC
UGL

FOUND CONC
UGL

Figure F11

RDX

TARGET CONC
UGL

FOUND CONC
UGL

Figure F12

TNT
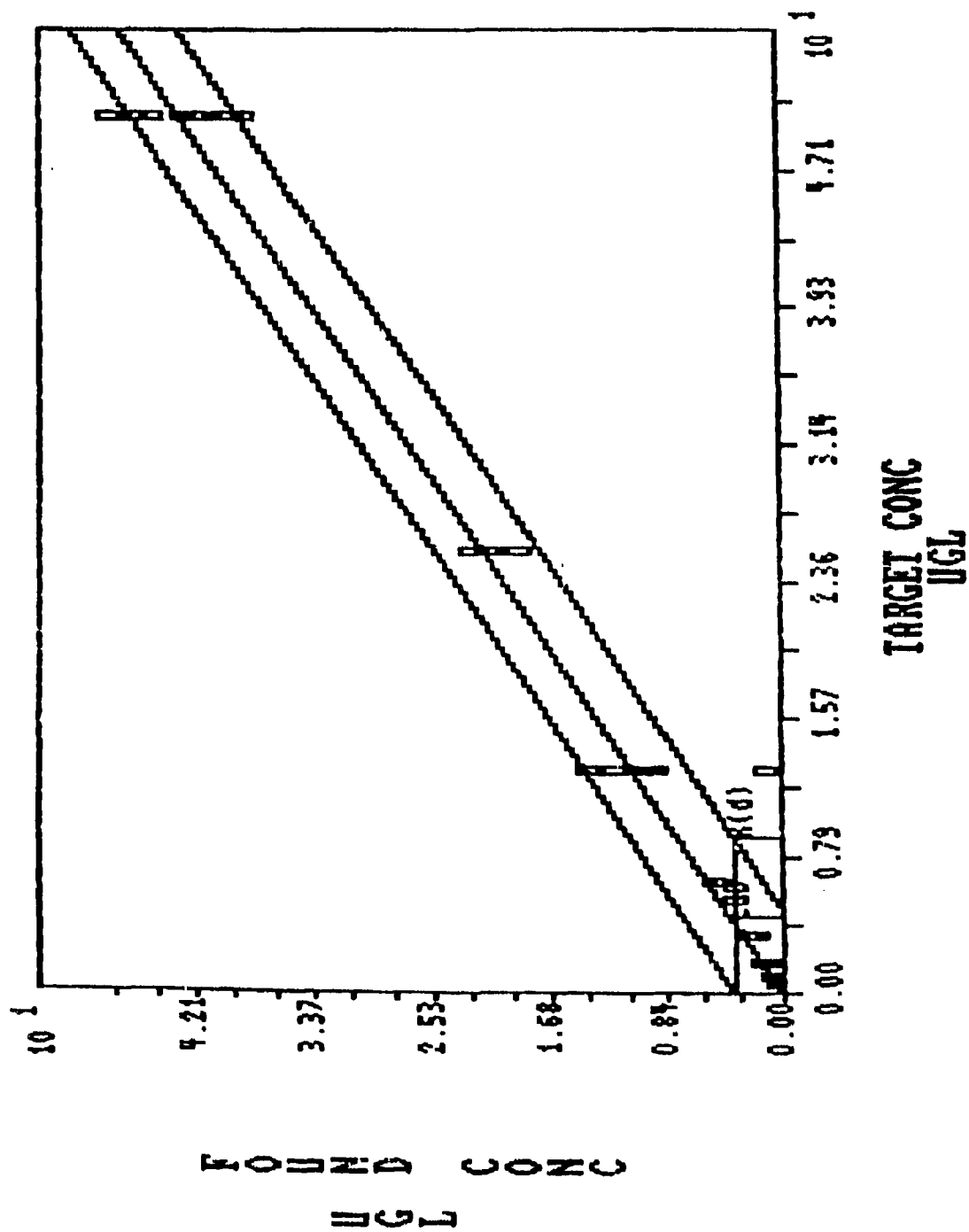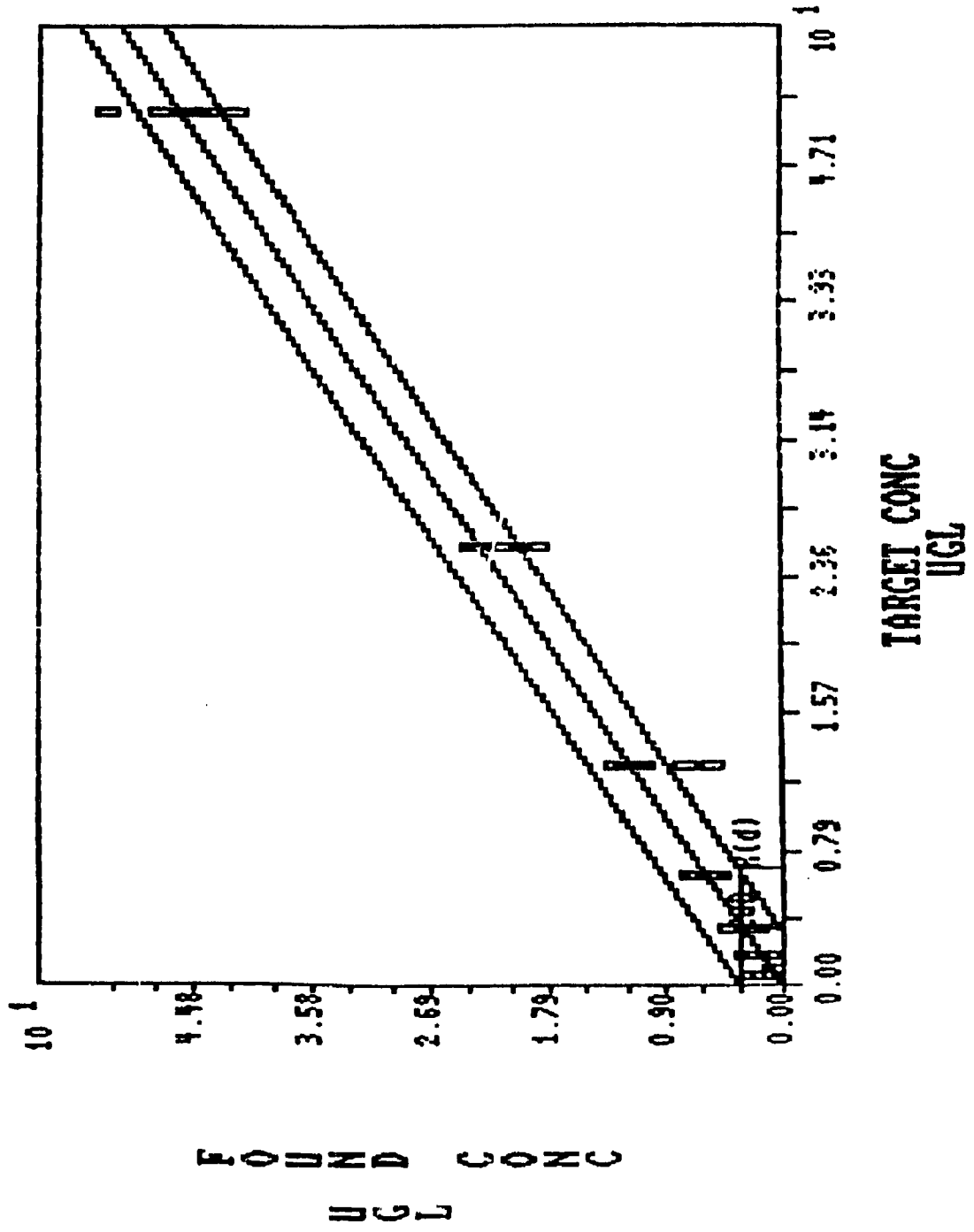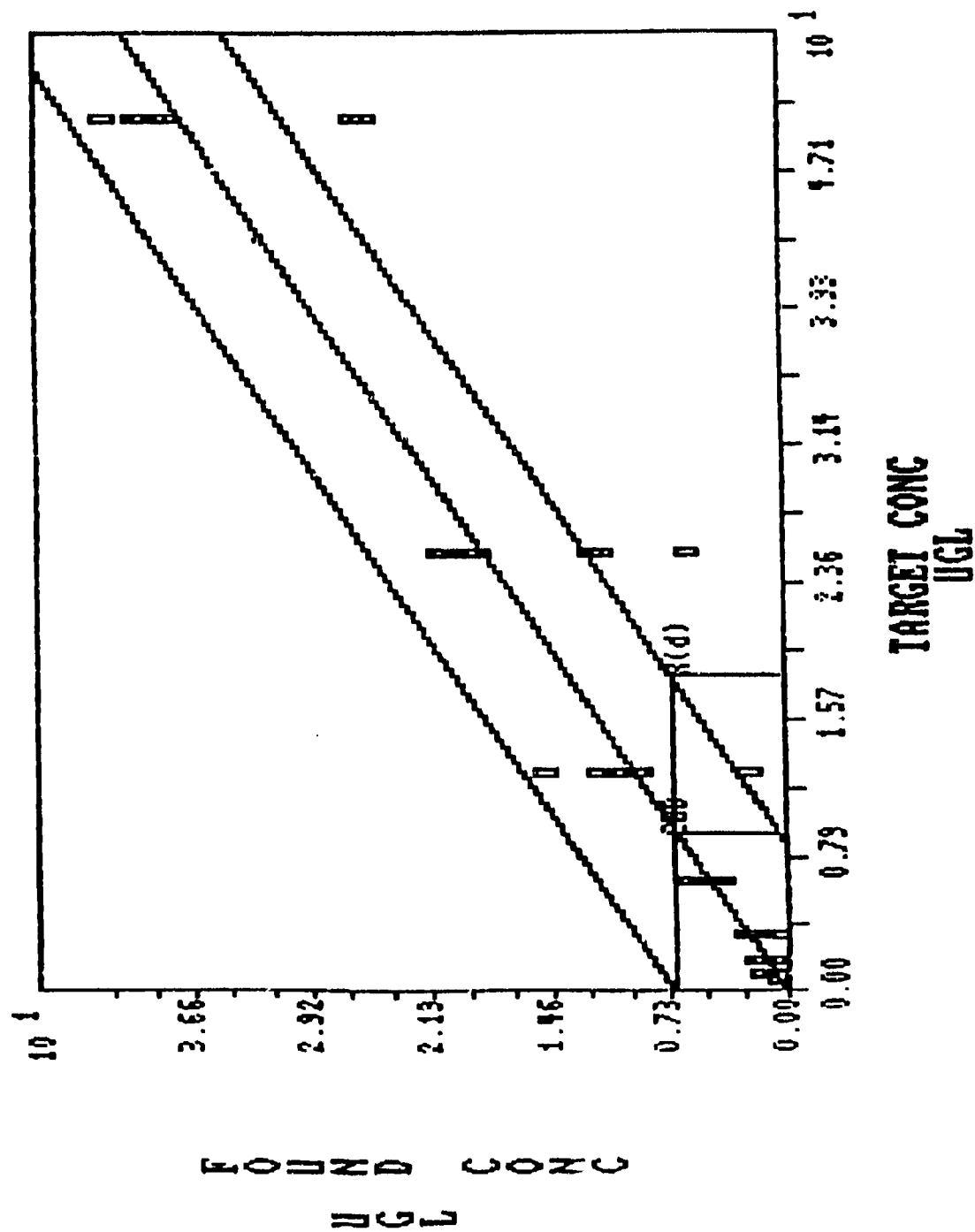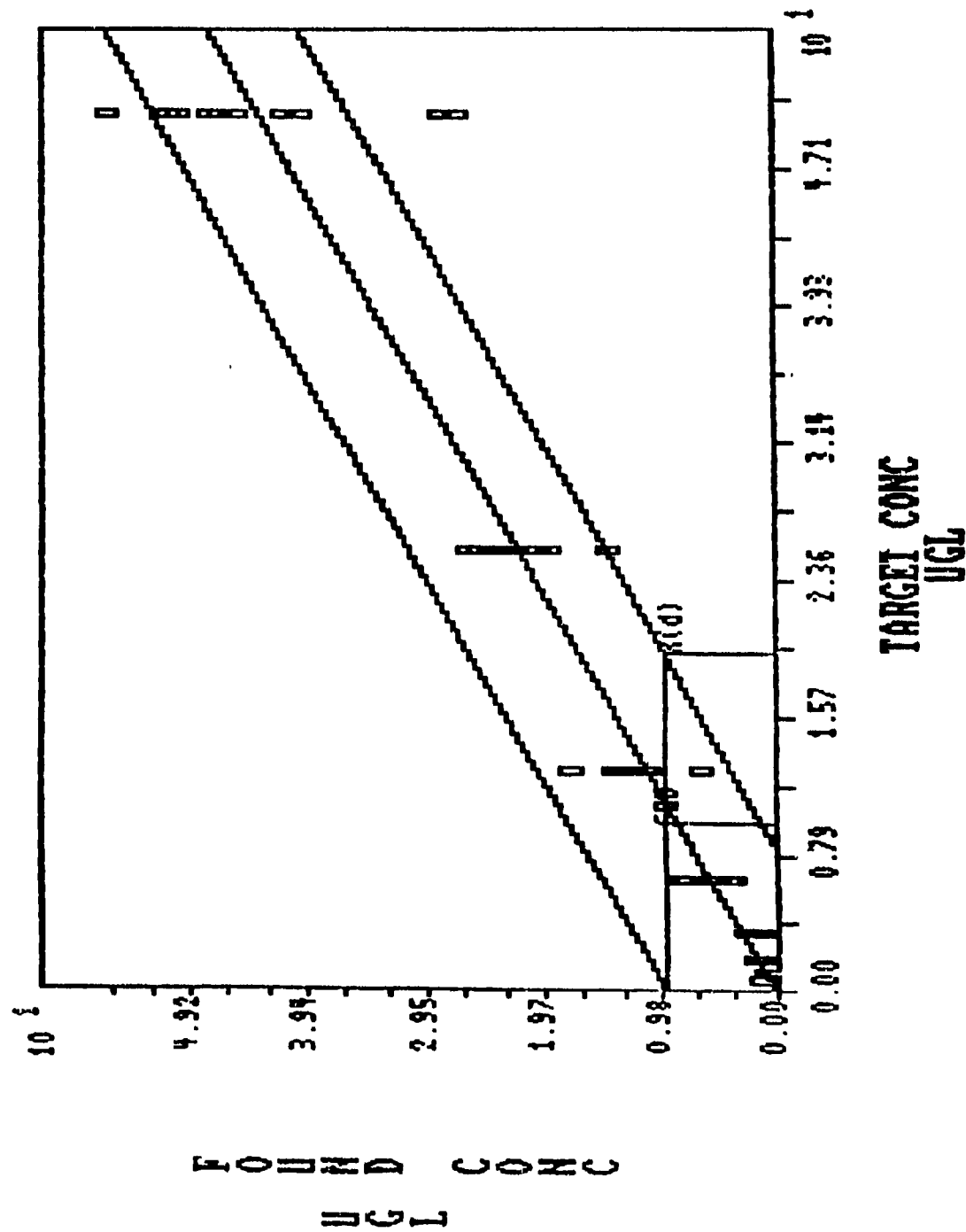
Figure F 13

2,4DNT

Figure F14

2,6DNT

TARGET CONC
ug/L

FOUND CONC
ug/L

Figure F15

2-AM

TARGET CONC
UGL

FOUND CONC
UGL

Figure F16

4-AM

TARGET CONC
UGL
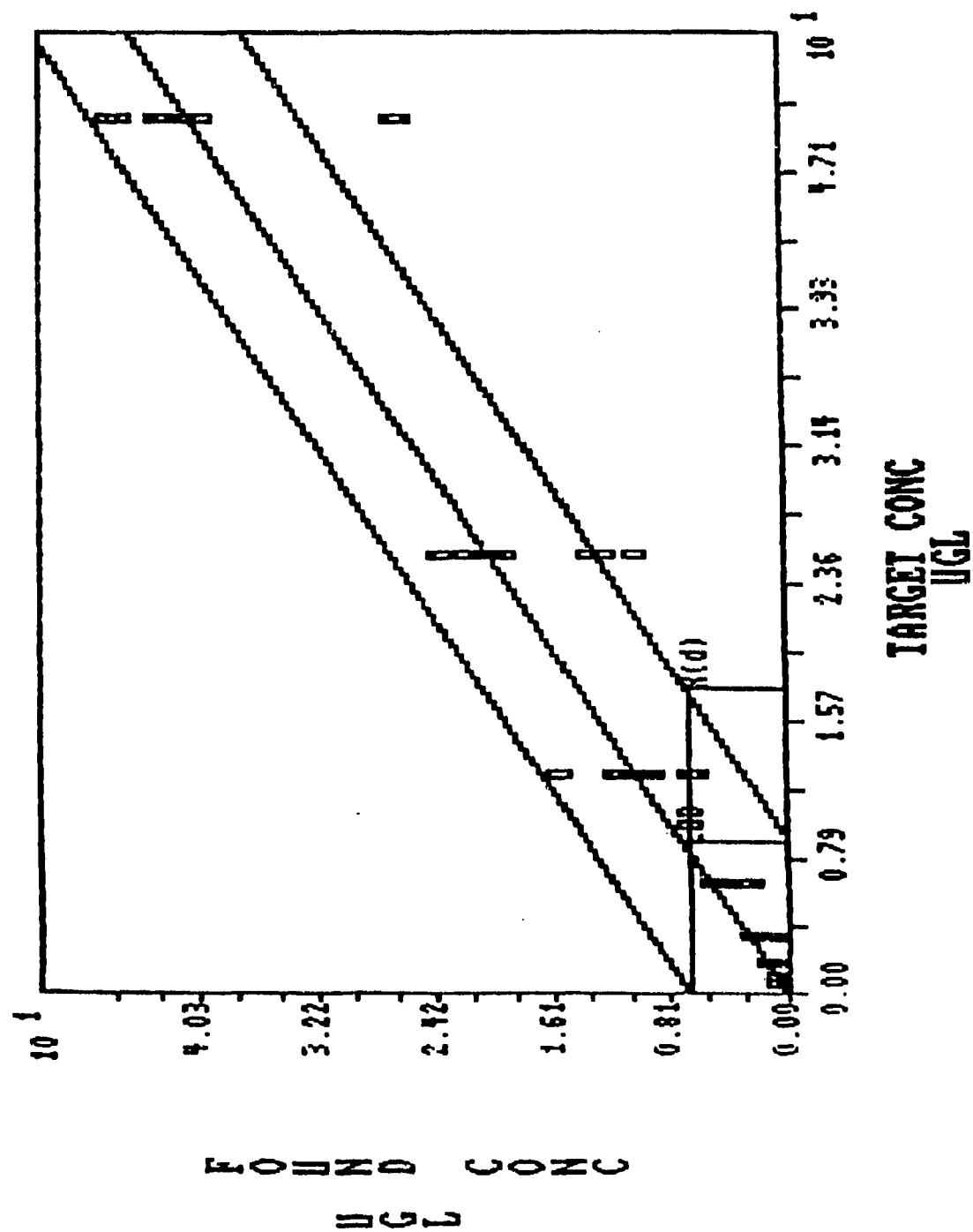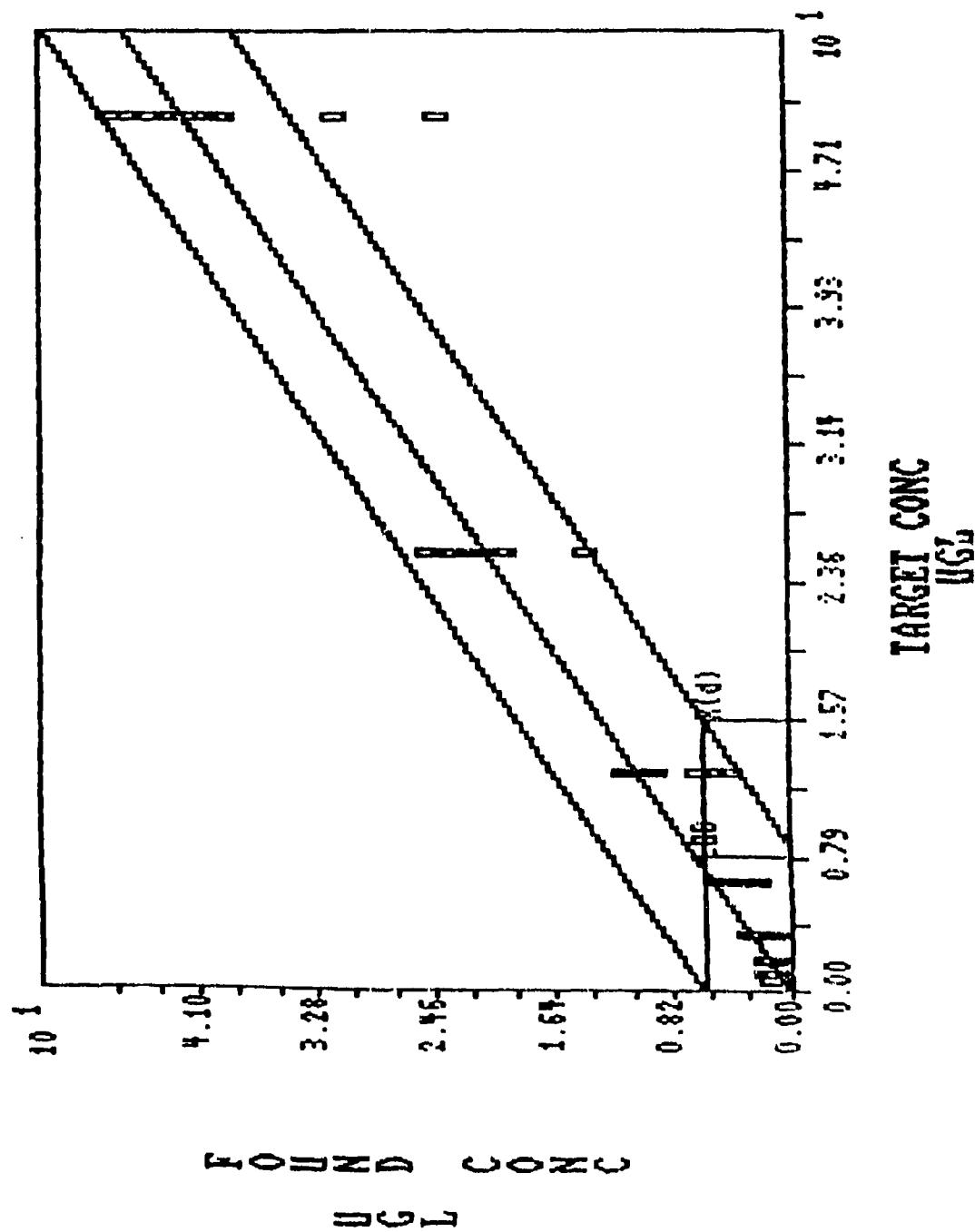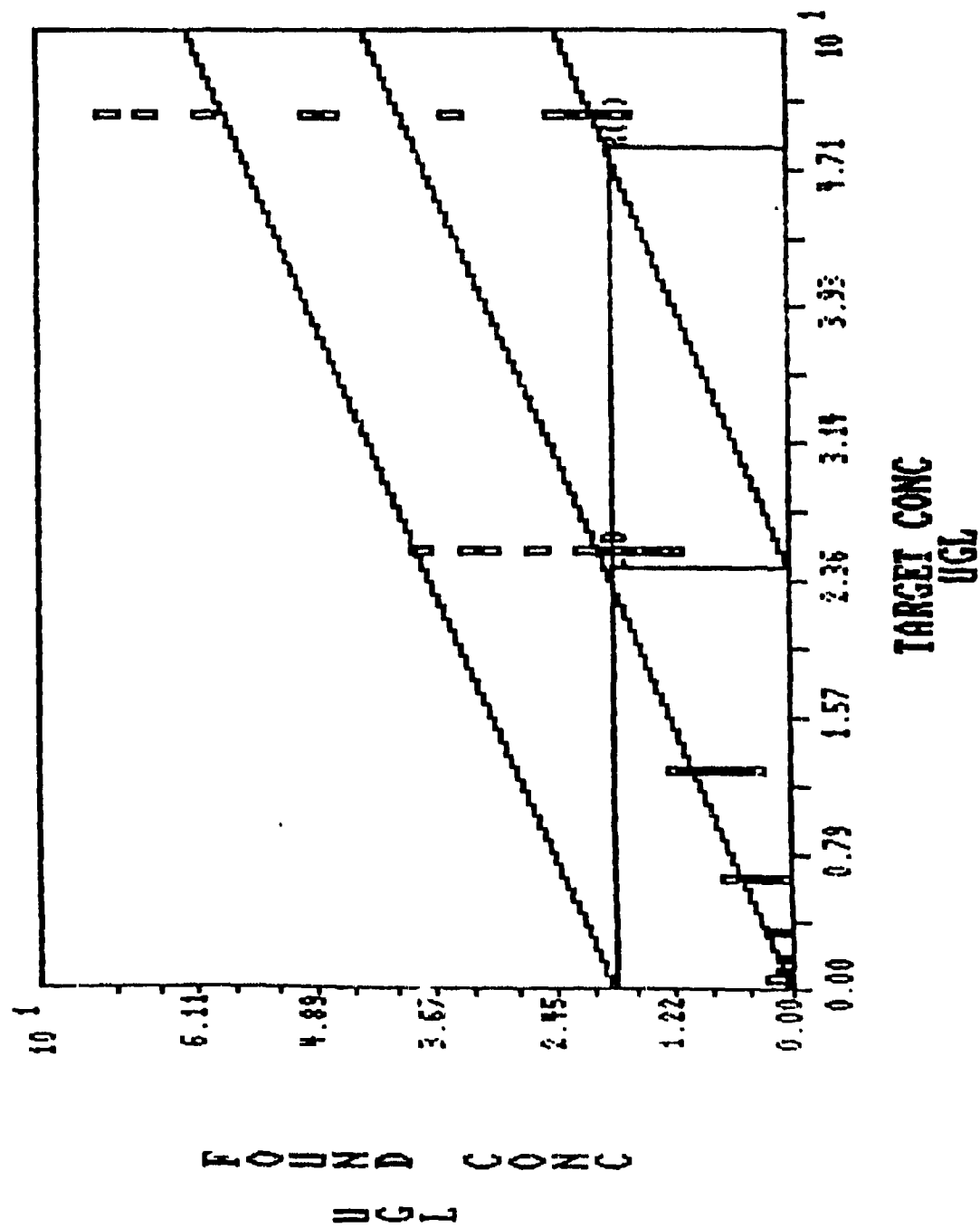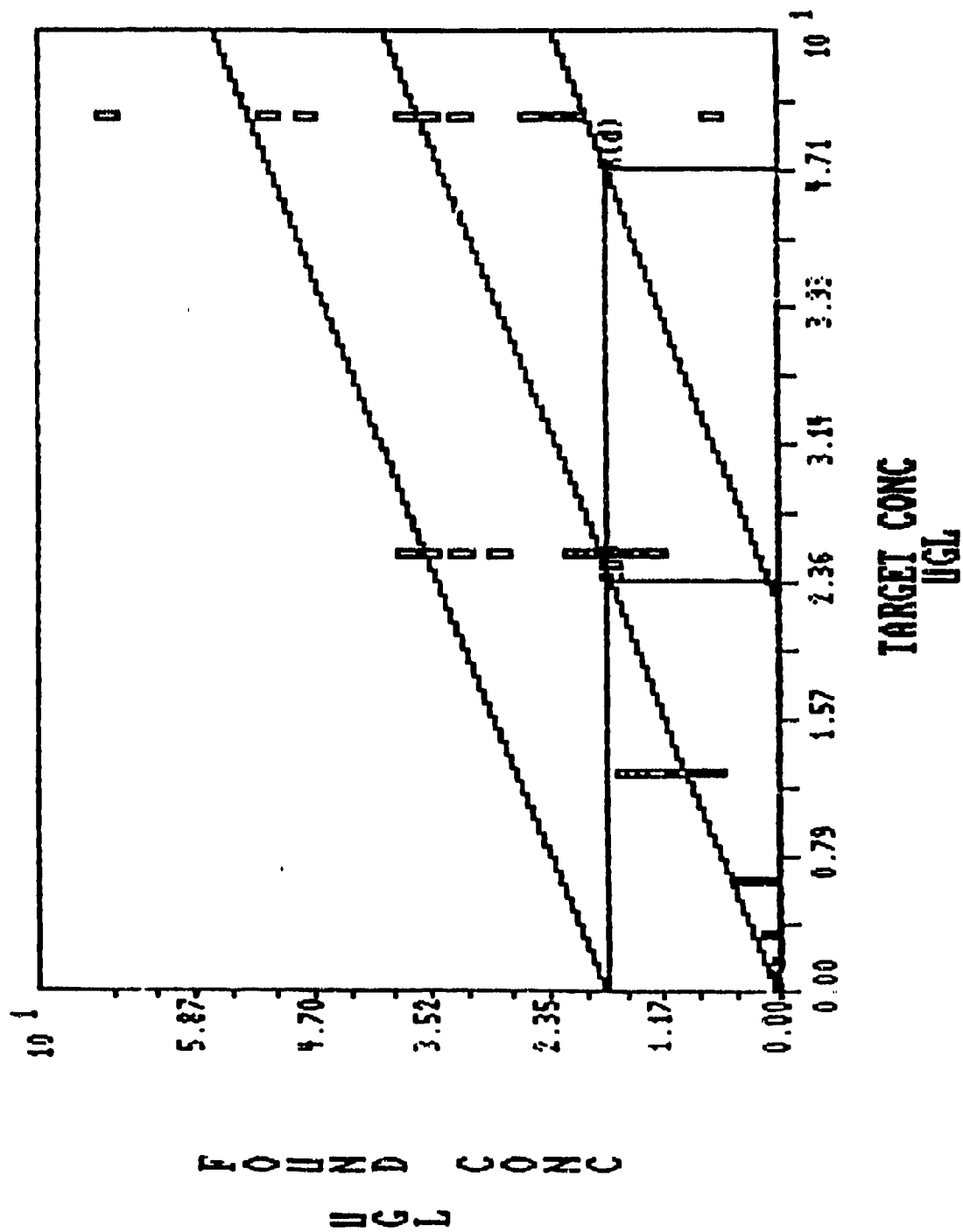
TABLE F9
CRITERION OF DETECTION FROM SOIL (mg/kg)

COMPOUNDS	CD
HMX	2.9
TNB	2.4
RDX	5.8
TNT	6.1
2,4 DNT	5.7
2,6 DNT	5.2
2-AM	15.4
4-AM	14.6

CERTIFICATION ANALYSIS   Report Date: 10/18/93
....

Method Name: HMX   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: HMX   Analysis Date   01/23/91
                                                Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
$Y = (-0.00399784) + (1.017741420)X$ $Y = (1.017141800)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.306303214	38	0.008060611	0.306750748	39	0.007865404
Total Error:	0.292517170	30	0.009750572	0.292517170	30	0.009750572
Lack of Fit:	0.013786044	8	0.001723255	0.014233578	9	0.001581509

LOF F-Ratio(F): 0.176733779   LOF F-Ratio(F): 0.162196496
Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

....
Zero Intercept Accepted   Calculated F: 0.055521102   Critical 95% F: 4.17
....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

Target Value Found Concentration

	Target Value	Found Concentration			
1:	10	9.8800000	9.9800000	10.410000	10.370000
2:	5	4.9900000	5.0200000	5.2000000	5.2000000
3:	2.5000000	2.5000000	2.5100000	2.5800000	2.5600000
4:	1.2500000	1.2500000	1.4600000	1.2600000	1.3000000
5:	0.6300000	0.6400000	0.6200000	0.6300000	0.6400000
6:	0.3200000	0.3400000	0.3100000	0.2900000	0.2900000
7:	0.1600000	0.1600000	0.1600000	0.1400000	0.1600000
8:	0.0800000	0.0900000	0.0600000	0.0600000	0.0690000
9:	0.0400000	0.0500000	0.0100000	0.0240000	0.0270000
10:	0.0200000	0.0040000	0.0080000	0.0050000	0.0024000

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: RADFORD   Units of Measure: UGG
Method Number: 1   Laboratory: MM
Compound: HMX   Analysis Date   12/31/91
                                       Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

              --- Model with Intercept ---   · Model through the Origin -
          Y = (-0.00458677) + (1.017921390)X   Y = (1.017233440)X

                  (SS)   (df)   (MS)   (SS)   (df)   (MS)
Residual: 0.308793193   38   0.008126137   0.309382294   39   0.007932879
otal Error: 0.294318503   30   0.009810617   0.294318503   30   0.009810617
Lack of Fit: 0.014474690   8   0.001809336   0.015063791   9   0.001673755

              LOF F-Ratio(F): 0.184426351   LOF F-Ratio(F): 0.170606456
              Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS
. . . .
Zero Intercept Accepted   Calculated F: 0.072494597 Critical 95% F: 4.17
. . . .

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value   Found Concentration

1:      0.0200000   0.0040000   0.0080000   0.0050000   +2.40E-04
2:      0.0400000   0.0500000   0.0100000   0.0240000   0.0270000
3:      0.0800000   0.0900000   0.0600000   0.0600000   0.0690000
4:      0.1600000   0.1600000   0.1600000   0.1400000   0.1600000
5:      0.3200000   0.3400000   0.3100000   0.2900000   0.2900000
6:      0.6300000   0.6400000   0.6200000   0.6300000   0.6400000
7:      1.2500000   1.2500000   1.4600000   1.2600000   1.3000000
8:      2.5000000   2.5800000   2.5800000   2.5000000   2.5100000
9:      5   4.9900000   5.0200000   5.2000000   5.2000000
10:     10   9.8800000   9.9800000   10.410000   10.370000

    *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/12/93

. . . .

Method Name:	MILAN	Units of Measure:	UGG
Method Number:		Laboratory:	MA
Compound:	TNB	Analysis Date	01/23/91
		Matrix:	WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---
$Y = (-0.04333250) + (1.013886250)X$

- Model through the Origin -
$Y = (1.007386980)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.293087156	38	0.007712820	0.345665012	39	0.008863205
Total Error:	0.217518860	30	0.007250629	0.217518860	30	0.007250629
Lack of Fit:	0.075568296	8	0.009446037	0.128146152	9	0.014238461

LOF F-Ratio(F): 1.302788687
Critical 95% F: 2.27

LOF F-Ratio(F): 1.963755419
Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

. . . .

**Zero Intercept Rejected Calculated F: 6.816943312 Critical 95% F: 4.17

. . . .

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

Target Value Found Concentration

	Target Value	Found Concentration			
1:	10	10.310000	10.360000	9.9700000	9.9700000
2:	5	4.8600000	4.9300000	5.0100000	5
3:	2.5000000	2.4200000	2.3200000	2.4900000	2.4800000
4:	1.2500000	1.0400000	1.2400000	1.2400000	1.2600000
5:	0.6300000	0.5900000	0.5800000	0.6100000	0.6300000
6:	0.3200000	0.2100000	0.1900000	0.3200000	0.3100000
7:	0.1600000	0.1600000	0.1500000	0.1600000	0.1600000
8:	0.0800000	0.0420000	0.0350000	0.0740000	0.0860000
9:	0.0400000	0.0500000	0.0100000	0.0240000	0.0270000
10:	0.0200000	0.0092000	0.0074000	0.0180000	0.0250000

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93

....

Method Name: RADFORD   Units of Measure: UGC
Method Number: 1   Laboratory: MM
Compound: TNB   Analysis Date   12/31/91
                                           Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

        --- Model with Intercept ---   - Model through the Origin -
     Y = (-0.04162067) + (1.014855330)X   Y = (1.008612820)X

                (SS)   (df)   (MS)   (SS)   (df) (MS)
Residual: 0.257655922   38 0.006780419 0.306161703 39 0.007850300
Total Error: 0.204409860   30 0.006813662 0.204409860 30 0.006813662
Lack of Fit: 0.053246062   8 0.006655758 0.101751843   9 0.011305760

        LOF F-Ratio(F): 0.976825347   LOF F-Ratio(F): 1.659278129
        Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

....
**Zero Intercept Rejected Calculated F: 7.153802884 Critical 95% F: 4.17
....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value   Found Concentration

 1:     0.0200000   0.0092000 0.0074000 0.0180000 0.0250000
 2:     0.0400000   0.0410000 0.0370000 0.0200000 0.0240000
 3:     0.0800000   0.0860000 0.0740000 0.0350000 0.0420000
 4:     0.1600000   0.0860000 0.0890000 0.1600000 0.1600000
 5:     0.3200000   0.2100000 0.1900000 0.3200000 0.3100000
 6:     0.6300000   0.6300000 0.6100000 0.5800000 0.5900000
 7:     1.2500000   1.0400000 1.2400000 1.2400000 1.2600000
 8:     2.5000000   2.4800000 2.4900000 2.5100000 2.5000000
 9:     5   4.8600000 4.9300000 5.0100000 5
10:    10   9.9700000 9.9700000 10.310000 10.360000

        ***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS

```
----------------------------
```

Method Name:	RDX	
Method Number:		
Compound:	RDX	

Units of Measure:	UGG
Laboratory:	MA
Analysis Date	01/23/91
Matrix:	WA

ANALYSIS OF RESIDUAL VARIATIONS

··· Model with Intercept ···   - Model through the Origin -
$Y = (-0.01057644) + (1.008113220)X$ $Y = (1.006526900)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.059129398	38	0.001556037	0.062261634	39	0.001596452
Total Error:	0.035166500	30	0.001172217	0.035166500	30	0.001172217
Lack of Fit:	0.023962899	8	0.002995362	0.027095134	9	0.003010570

LOF F-Ratio(F): 2.555297495   LOF F-Ratio(F): 2.568271338
Critical 95% F: 2.27   Critical 95% F: 2.21
Data Not Linear   Data Not Linear

ZERO INTERCEPT HYPOTHESIS

```
------------------------
```
** Models not linear.   Do not test Zero Intercept hypothesis.
```
------------------------
```
Diagnose and correct analytical system before continuing.

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

Target Value   Found Concentration

	Target Value	Found Concentration			
1:	10	10.060000	10.150000	10.150000	10.060000
2:	5	4.8900000	4.9400000	5.0500000	5.0200000
3:	2.5000000	2.4400000	2.4700000	2.5100000	2.5200000
4:	1.2500000	1.2100000	1.2300000	1.2200000	1.2900000
5:	0.6300000	0.6300000	0.6100000	0.6200000	0.6200000
6:	0.3200000	0.3400000	0.3300000	0.3400000	0.3100000
7:	0.1600000	0.1600000	0.1500000	0.1700000	0.1900000
8:	0.0800000	0.0790000	0.0900000	0.0880000	0.1000000
9:	0.0400000	0.0230000	0.0310000	0.0310000	0.0500000
10:	0.0200000	0.0320000	0.0200000	0.0020000	0.0020000

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/19/93
- -

Method Name: RDX   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: RDX   Analysis Date   01/23/91
                                       Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

              --- Model with Intercept ---   - Model through the Origin -
          Y = (-0.01050523) + (1.008102610)X   Y = (1.006526980)X

              (SS)   (df)   (MS)   (SS)   (df)   (MS)
Residual: 0.059094913   38 0.001555129 0.062185112   39 0.001594490
Total Error: 0.035115500   30 0.001170517 0.035115500   30 0.001170517
Lack of Fit: 0.023979413   8 0.002997427 0.027069612   9 0.003007735

              LOF F-Ratio(F): 2.560772294   LOF F-Ratio(F): 2.569578676
              Critical 95% F: 2.27   Critical 95% F: 2.21
              Data Not Linear   Data Not Linear

                      ZERO INTERCEPT HYPOTHESIS
- -
** Models not linear.   Do not test Zero Intercept hypothesis.
- -
          Diagnose and correct analytical system before continuing.

                    ****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

          Target Value   Found Concentration

    1:    10   10.060000   10.150000   10.150000   10.060000
    2:    5   4.8900000   4.9400000   5.0500000   5.0200000
    3:    2.5000000   2.4400000   2.4700000   2.5100000   2.5200000
    4:    1.2500000   1.2100000   1.2300000   1.2200000   1.2900000
    5:    0.6300000   0.6300000   0.6100000   0.6200000   0.6200000
    6:    0.3200000   0.3400000   0.3300000   0.3400000   0.3100000
    7:    0.1600000   0.1600000   0.1500000   0.1700000   0.1900000
    8:    0.0800000   0.0790000   0.0900000   0.0880000   0.1000000
    9:    0.0400000   0.0230000   0.0310000   0.0310000   0.0500000
   10:    0.0200000   0.0320000   0.0200000   0.0020000   0.0020000

       ***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

Appendix B   96

CERTIFICATION ANALYSIS   Report Date: 10/12/93

- -

Method Name: RADFORD   Units of Measure: UGG
Method Number: 1   Laboratory: MM
Compound: RDX   Analysis Date   12/31/91
                                       Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

··· Model with Intercept ···   - Model through the Origin -
$Y = (0.013858142) + (1.001916230)X$ $Y = (1.003992260)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.373006260	34	0.010970772	0.377621272	35	0.010789179
Total Error:	0.226222000	27	0.008378593	0.226222000	27	0.008378593
Lack of Fit:	0.146784260	7	0.020969180	0.151399272	8	0.018924909

LOF F-Ratio(F): 2.502709109   LOF F-Ratio(F): 2.258721711
Critical 95% F: 2.37   Critical 95% F: 2.31
Data Not Linear

ZERO INTERCEPT HYPOTHESIS

- -
** Intercept model not linear.   Do not test Zero Intercept hypothesis.
- -
           Diagnose and correct analytical system before continuing.

****

TABLE OF DATA POINTS   Targets: 9   Measures per Target: 4

        Target Value   Found Concentration

#	Target Value	Found Concentration			
1:	0.0400000	0	0	0.0270000	0.0270000
2:	0.0800000	0	0.0580000	0.0600000	0.0600000
3:	0.1600000	0.1400000	0.2100000	0.1900000	0.1900000
4:	0.3200000	0.2600000	0.3900000	0.1900000	0.3400000
5:	0.6250000	0.6100000	0.6300000	0.5800000	0.5800000
6:	1.2500000	1.5000000	1.4000000	1.3000000	1.1000000
7:	2.5000000	2.6000000	2.5000000	2.8000000	2.8000000
8:	5	5.1000000	5.1000000	4.9000000	4.9000000
9:	10	10	10.010000	10	10

    *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/19/93

Method Name: TNT   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: TNT   Analysis Date   01/23/91
                                              Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
Y = (-0.01813630) + (1.007155650)X   Y = (1.004435460)X

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.113801306	38	0.002994771	0.123011.68	39	0.003154143
Total Error:	0.102973750	30	0.003432458	0.102973750	30	0.003432458
Lack of Fit:	0.010827556	8	0.001353444	0.020037838	9	0.002226426

LOF F-Ratio(F): 0.394307627   LOF F-Ratio(F): 0.648639030
Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

....
Zero Intercept Accepted   Calculated F: 3.075454301 Critical 95% F: 4.17
....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

Target Value Found Concentration

	Target Value	Found Concentration			
1:	10	9.9300000	10.110000	10.180000	10.080000
2:	5	4.8600000	4.8900000	5.1100000	5.0700000
3:	2.5000000	2.4600000	2.4400000	2.5200000	2.5500000
4:	1.2500000	1.1500000	1.2000000	1.2300000	1.2900000
5:	0.6300000	0.6200000	0.5900000	0.6400000	0.6200000
6:	0.3200000	0.3200000	0.2900000	0.3100000	0.3400000
7:	0.1600000	0.1400000	0.1400000	0.1600000	0.1800000
8:	0.0800000	0.0640000	0.0660000	0.0740000	0.0840000
9:	0.0400000	0.0280000	0.0280000	0.0270000	0.0260000
10:	0.0200000	0.0020000	0.0020000	0.0140000	0.0120000

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: RADFORD   Units of Measure: UGG
Method Number: 1   Laboratory: MM
Compound: TNT   Analysis Date   12/31/91
                                        Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

```
        ... Model with Intercept ---    - Model through the Origin -
        Y = (-0.01801080) + (1.007417900)X      Y = (1.004716530)X
```

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.110208263	38	0.002900217	0.119291521	39	0.003058757
Total Error:	0.101346750	30	0.003378225	0.101346750	30	0.003378225
Lack of Fit:	0.008861513	8	0.001107689	0.017944771	9	0.001993863

```
        LOF F-Ratio(F): 0.327890867   LOF F-Ratio(F): 0.590210375
        Critical 95% F: 2.27          Critical 95% F: 2.21
```


ZERO INTERCEPT HYPOTHESIS

. . . .
Zero Intercept Accepted   Calculated F: 3.131923094 Critical 95% F: 4.17
. . . .

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value   Found Concentration

No.	Target Value	Found Concentration			
1:	0.0200000	0	0	0.0140000	0.0120000
2:	0.0400000	0.0260000	0.0270000	0.0280000	0.0280000
3:	0.0800000	0.0640000	0.0660000	0.0740000	0.0840000
4:	0.1600000	0.1400000	0.1400000	0.1600000	0.1800000
5:	0.3200000	0.3400000	0.3100000	0.3200000	0.2900000
6:	0.6300000	0.6200000	0.5900000	0.6400000	0.6200000
7:	1.2500000	1.2900000	1.2300000	1.1500000	1.2000000
8:	2.5000000	2.4600000	2.4400000	2.5200000	2.5500000
9:	5	5.0700000	5.1100000	4.8400000	4.9400000
10:	10	9.9300000	10.110000	10.180000	10.080000

        *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS   Report Date: 10/18/93
....

Method Name: 2,4   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: 2,4   Analysis Date   01/23/91
                                         Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

          ··· Model with Intercept ···   - Model through the Origin -
      Y = (-0.02530612) + (1.025863060)X   Y = (1.022067500)X

             (SS)   (df) (MS)   (SS)   (df) (MS)
Residual: 0.405071561 38   0.010659778   0.423003471 39   0.010846243
Total Error: 0.360487280 30   0.012016243   0.360487280 30   0.012016243
Lack of Fit: 0.044584281   8   0.005573035   0.062516191   9   0.006946243

          LOF F-Ratio(F): 0.463791826   LOF F-Ratio(F): 0.578071169
          Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS
....
Zero Intercept Accepted   Calculated F: 1.682202963 Critical 95% F: 4.17
....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value Found Concentration

   1:    10   10.090000 10.110000 10.560000 10.360000
   2:    5   4.8400000 4.8500000 5.2500000 5.2000000
   3:    2.5000000   2.3700000 2.3900000 2.5700000 2.6300000
   4:    1.2500000   1.2000000 1.2000000 1.2600000 1.3100000
   5:    0.6300000   0.6200000 0.5900000 0.6500000 0.6700000
   6:    0.3200000   0.3400000 0.3400000 0.3100000 0.3100000
   7:    0.1600000   0.1500000 0.1500000 0.1600000 0.1900000
   8:    0.0800000   0.0730000 0.0720000 0.0800000 0.0730000
   9:    0.0400000   0.0220000 0.0140000 0.0088000 0.0360000
  10:    0.0200000   0.0020000 0.0020000 0.0020000 0.0020000

    *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Method Name: RADFORD
Method Number: 1
Compound: 2-4DNT

Units of Measure: UGG
Laboratory: MM
Analysis Date   12/31/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

	--- Model with Intercept --- $Y = (-0.02459154) + (1.023768270)X$			- Model through the Origin - $Y = (1.020079880)X$		
	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.460537936	38	0.012119419	0.477471436	39	0.012242857
Total Error:	0.416563030	30	0.013885434	0.416563030	30	0.013885434
Lack of Fit:	0.043974906	8	0.005496863	0.060908406	9	0.006767601

LOF F-Ratio(F): 0.395872619   LOF F-Ratio(F): 0.487388475
Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted   Calculated F: 1.397220402   Critical 95% F: 4.17

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

	Target Value	Found Concentration			
1:	0.0200000	0	0	0	0
2:	0.0400000	0.0220000	0.0140000	0.0088000	0.0360000
3:	0.0800000	0.0730000	0.0800000	0.0720000	0.0730000
4:	0.1600000	0.1500000	0.1500000	0.1600000	0.1900000
5:	0.3200000	0.3100000	0.3100000	0.3400000	0.3400000
6:	0.6300000	0.6200000	0.5900000	0.6500000	0.6700000
7:	1.2500000	1.2000000	1.2000000	1.2600000	1.3100000
8:	2.5000000	2.3700000	2.3900000	2.5700000	2.6300000
9:	5	4.8400000	4.8000000	5.2500000	5.2000000
10:	10	10.009000	10.110000	10.560000	10.360000

***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS

Report Date: 10/18/93

- -

Method Name:	2,6	Units of Measure:	UGG
Method Number:		Laboratory:	MA
Compound:	2,6	Analysis Date	01/23/91
		Matrix:	WA

ANALYSIS OF RESIDUAL VARIATIONS

··· Model with Intercept ···   - Model through the Origin -

$Y = (-0.03122974) + (1.047214870)X$ $Y = (1.042530850)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	1.981234090	38	0.052137739	2.008543500	39	0.051501115
Total Error:	1.940400000	30	0.064680000	1.940400000	30	0.064680000
Lack of Fit:	0.040834090	8	0.005104261	0.068143500	9	0.007571500

LOF F-Ratio(F): 0.078915604   LOF F-Ratio(F): 0.117060915

Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

- -

Zero Intercept Accepted   Calculated F: 0.523793521 Critical 95% F: 4.17

- -

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

	Target Value	Found Concentration			
1:	10	10.140000	9.8100000	11.240000	10.730000
2:	5	4.7800000	4.8000000	5.5700000	5.4600000
3:	2.5000000	2.3200000	2.3200000	2.6800000	2.7700000
4:	1.2500000	1.3800000	1.2900000	1.2600000	1.2100000
5:	0.6300000	0.6000000	0.5900000	0.6800000	0.7100000
6:	0.3200000	0.3200000	0.2800000	0.3500000	0.3700000
7:	0.1600000	0.1700000	0.2100000	0.1400000	0.1200000
8:	0.0800000	0.0590000	0.0460000	0.0800000	0.0430000
9:	0.0200000	0	0	0	0
10:	0.0400000	0	0	0	0

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 15b

CERTIFICATION ANALYSIS   Report Date: 10/12/93
....

Method Name: RADFORD   Units of Measure: UGG
Method Number: 1   Laboratory: MM
Compound: 2-6DNT   Analysis Date   12/31/91
                                        Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

··· Model with Intercept ···   · Model through the Origin -
Y = (-0.03122974) + (1.047214870)X   Y = (1.042530850)X

             (SS)   (df)   (MS)   (SS)   (df) (MS)
Residual: 1.981234090 38   0.052137739   2.008543500 39   0.051501115
Total Error: 1.940400000 30   0.064680000   1.940400000 30   0.064680000
Lack of Fit: 0.040834090 8   0.005104261   0.068143500 9   0.007571500

        LOF F-Ratio(F): 0.078915604   LOF F-Ratio(F): 0.117060915
        Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS
....
Zero Intercept Accepted   Calculated F: 0.523793521 Critical 95% F: 4.17
....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value Found Concentration

 1:     0.0200000   0   0   0   0
 2:     0.0400000   0   0   0   0
 3:     0.0800000   0.0460000 0.0590000 0.0800000 0.0430000
 4:     0.1600000   0.1200000 0.1400000 0.1700000 0.2100000
 5:     0.3200000   0.3200000 0.2800000 0.3500000 0.3700000
 6:     0.6300000   0.7100000 0.6800000 0.5900000 0.6000000
 7:     1.2500000   1.2600000 1.2100000 1.2900000 1.3800000
 8:     2.5000000   2.7700000 2.6800000 2.3200000 2.3200000
 9:     5   4.7800000 4.8000000 5.5700000 5.4600000
10:     10   10.140000 9.8100000 11.240000 10.730000

   ***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

Appendix B   103

CERTIFICATION ANALYSIS   Report Date: 10/18/93
. . . .

Method Name: 2AM   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: 2AM   Analysis Date   01/23/91
                                         Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

           ... Model with Intercept ...   - Model through the Origin -
        Y = (-0.04092383) + (1.009736910)X   Y = (1.003598910)X

              (SS)   (df)   (MS)   (SS)   (df)   (MS)
Residual: 0.263036377 38   0.006922010   0.309931526 39   0.007946962
Total Error: 0.218409500 30   0.007280317   0.218409500 30   0.007280317
Lack of Fit: 0.044626877   8   0.005578360   0.091522026   9   0.010169114

              LOF F-Ratio(F): 0.766224861   LOF F-Ratio(F): 1.396795561
              Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS
. . . .
**Zero Intercept Rejected Calculated F: 6.774787892 Critical 95% F: 4.17
. . . .

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value   Found Concentration

1:	10	9.9800000	10.030000	10.430000	9.9500000
2:	5	4.8300000	5.0500000	4.9200000	5.0400000
3:	2.5000000	2.3800000	2.4100000	2.4900000	2.4100000
4:	1.2500000	1.1900000	1.1700000	1.2100000	1.1700000
5:	0.6300000	0.6200000	0.5700000	0.5600000	0.7100000
6:	0.3200000	0.2500000	0.3300000	0.3400000	0.3300000
7:	0.1600000	0.1400000	0.1800000	0.1400000	0.0750000
8:	0.0800000	0.0430000	0.0800000	0.0230000	0.0560000
9:	0.0400000	0.0190000	0.0020000	0.0040000	0.0020000
10:	0.0200000	0.0020000	0.0020000	0.0020000	0.0020000

        ***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

Appendix B   104

CERTIFICATION ANALYSIS   Report Date: 10/12/93
. . . .

Method Name: RADFORD   Units of Measure: UGG
Method Number: 1   Laboratory: MM
Compound: 2AMDNT   Analysis Date   12/31/91
                                             Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   - Model through the Origin -
$Y = (-0.04248105) + (1.009965530)X$ $Y = (1.003593960)X$

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.262130576	38	0.006898173	0.312662515	39	0.008016988
Total Error:	0.218400500	30	0.007280017	0.218400500	30	0.007280017
Lack of Fit:	0.043730076	8	0.005466260	0.094262015	9	0.010473557

LOF F-Ratio(F): 0.750858102   LOF F-Ratio(F): 1.438672149
Critical 95% F: 2.27   Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS
. . . .
**Zero Intercept Rejected   Calculated F: 7.325409005   Critical 95% F: 4.17
. . . .

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value Found Concentration

	Target Value	Found Concentration			
1:	0.0200000	·0	0	0	0
2:	0.0400000	0.0190000	0	0	0
3:	0.0800000	0.0560000	0.0230000	0.0230000	0.0710000
4:	0.1600000	0.0760000	0.1400000	0.1400000	0.1800000
5:	0.3200000	0.3400000	0.3300000	0.3300000	0.2500000
6:	0.6300000	0.6200000	0.5700000	0.5600000	0.7100000
7:	1.2500000	1.1900000	1.1700000	1.1700000	1.2100000
8:	2.5000000	2.3800000	2.4100000	2.4100000	2.4900000
9:	5	4.8300000	5.0500000	5.0400000	4.9200000
10:	10	9.9800000	10.030000	10.430000	9.9500000

***   END OF CERTIFICATION LACK OF FIT DATA TABLE   ***

CERTIFICATION ANALYSIS   Report Date: 10/18/93
- -

Method Name: MILAN   Units of Measure: UGG
Method Number: Laboratory: MA
Compound: 4AMDNT   Analysis Date   01/23/91
                                                 Matrix: WA

ANALYSIS OF RESIDUAL VARIATION'

	--- Model with Intercept ---			- Model through the Origin -		
	$Y = (-0.05365346) + (1.006851730)X$			$Y = (0.998804462)X$		
	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.181320988	38	0.004771605	0.261927629	39	0.006716093
Total Error:	0.138595000	30	0.004619833	0.138595000	30	0.004619833
Lack of Fit:	0.042725988	8	0.005340748	0.123332629	9	0.013703625

          LOF F-Ratio(F): 1.156047873   LOF F-Ratio(F): 2.966259702
          Critical 95% F: 2.27   Critical 95% F: 2.21
                                          Data Not Linear

ZERO INTERCEPT HYPOTHESIS
- -
Zero Intercept Rejected Calculated F: 16.89298295 Critical 95% F: 4.17
Model not linear through Origin
- -

                    ****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

        Target Value   Found Concentration

	Target Value	Found Concentration			
1:	10	10.030000	10	10.160000	9.9900000
2:	5	4.8700000	4.8500000	4.9100000	5.1000000
3:	2.5000000	2.3800000	2.4000000	2.4900000	2.4900000
4:	1.2500000	1.2100000	1.1600000	1.2200000	1.2100000
5:	0.6300000	0.6000000	0.6100000	0.5800000	0.6500000
6:	0.3200000	0.3500000	0.3200000	0.2100000	0.0360000
7:	0.1600000	0.0600000	0.0650000	0.1100000	0.0810000
8:	0.0800000	0.0210000	0.0320000	0.0360000	0.0210000
9:	0.0400000	0.0830000	0.0360000	0.0210000	0.0020000
10:	0.0200000	0.0020000	0.0020000	0.0020000	0.0020000

    *** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/12/93

....

Method Name:	RADFORD	Units of Measure:	UGG
Method Number:	1	Laboratory:	MM
Compound:	4AMDNT	Analysis Date	12/31/91
		Matrix:	WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---   · Model through the Origin ·
Y = (-0.05243419) + (1.006758340)X   Y = (0.998893951)X

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.134476662	38	0.003538860	0.211461379	39	0.005422087
Total Error:	0.106517568	30	0.003550586	0.106517568	30	0.003550586
Lack of Fit:	0.027959094	8	0.003494887	0.104943811	9	0.011660423

LOF F-Ratio(F): 0.984312771   LOF F-Ratio(F): 3.284084587
Critical 95% F: 2.27   Critical 95% F: 2.21
                                Data Not Linear

ZERO INTERCEPT HYPOTHESIS

....

Zero Intercept Rejected Calculated F: 21.75410367   Critical 95% F: 4.17
Model not linear through Origin

....

****

TABLE OF DATA POINTS   Targets: 10   Measures per Target: 4

Target Value   Found Concentration

	Target Value				
1:	0.0200000	0	0	0	0
2:	0.0400000	0.0083000	0	0	0.0190000
3:	0.0800000	0.0210000	0.0360000	0.0320000	0.0210000
4:	0.1600000	0.0650000	0.0600000	0.1100000	0.0810000
5:	0.3200000	0.1200000	0.2900000	0.3200000	0.3500000
6:	0.6300000	0.6100000	0.6000000	0.5800000	0.6500000
7:	1.2500000	1.2200000	1.2100000	1.1600000	1.2100000
8:	2.5000000	2.3800000	2.4000000	2.4900000	2.4900000
9:	5	4.8700000	4.8500000	5.1000000	4.9100000
10:	10	10.030000	10	10.160000	9.9900000

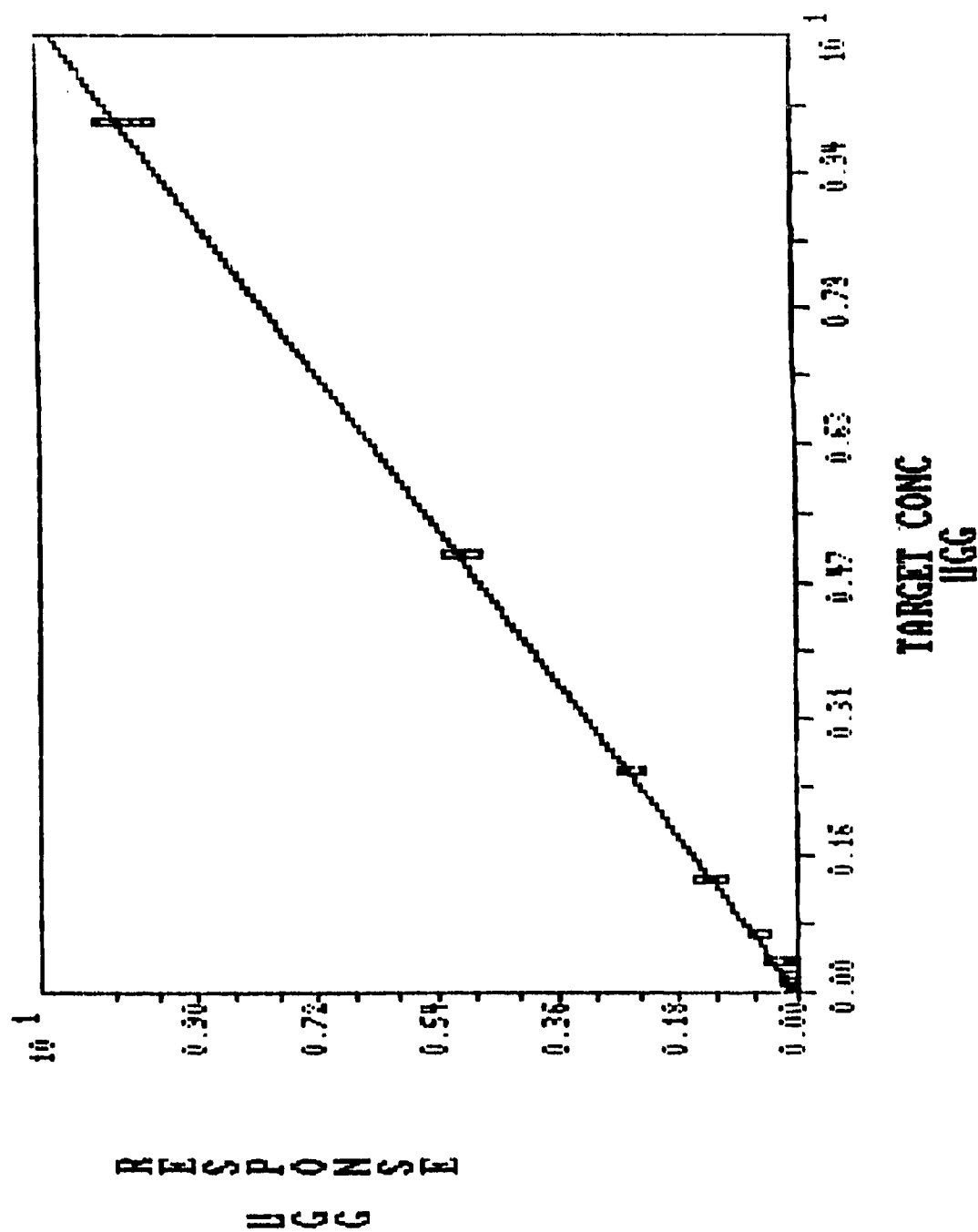*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***
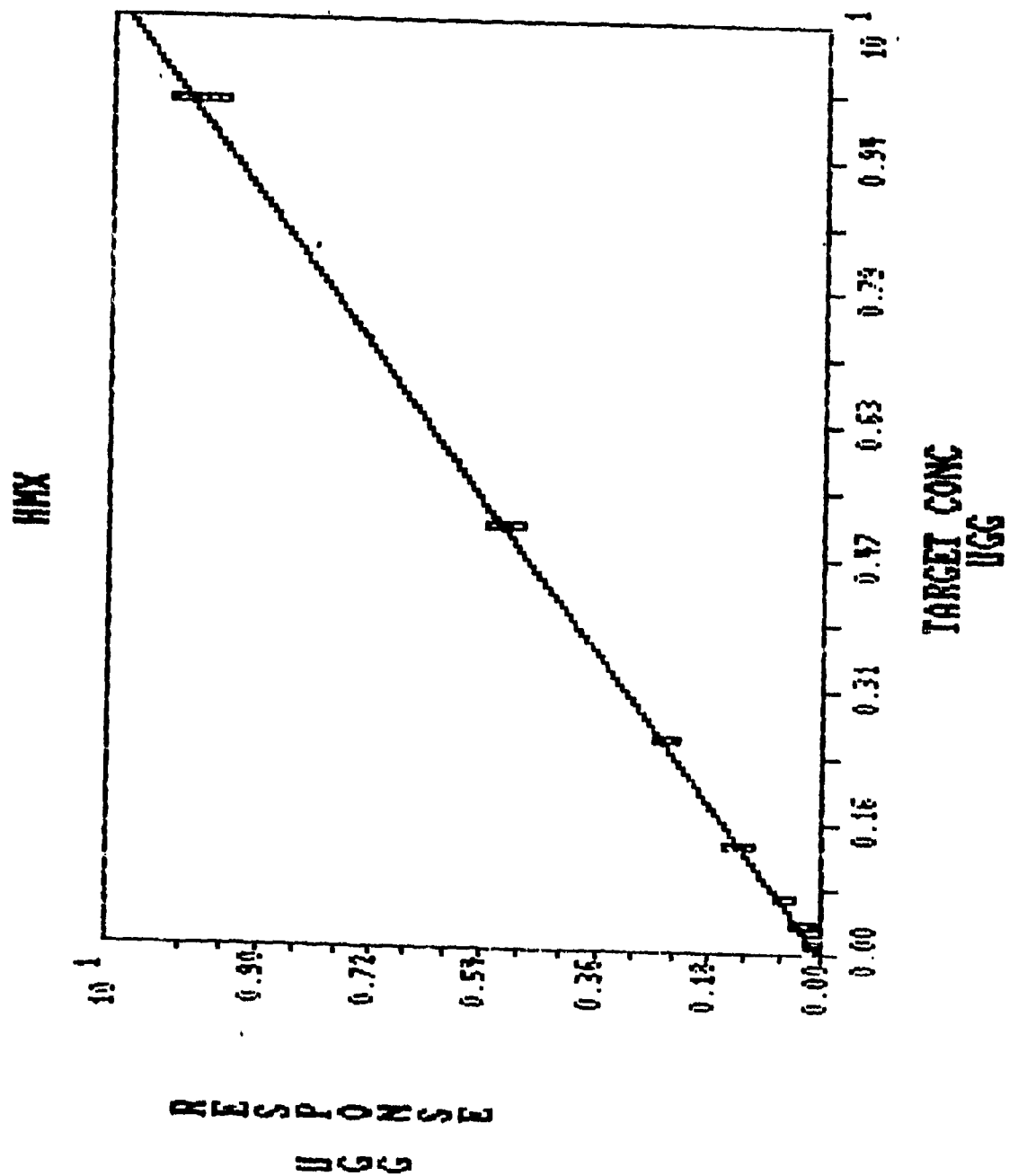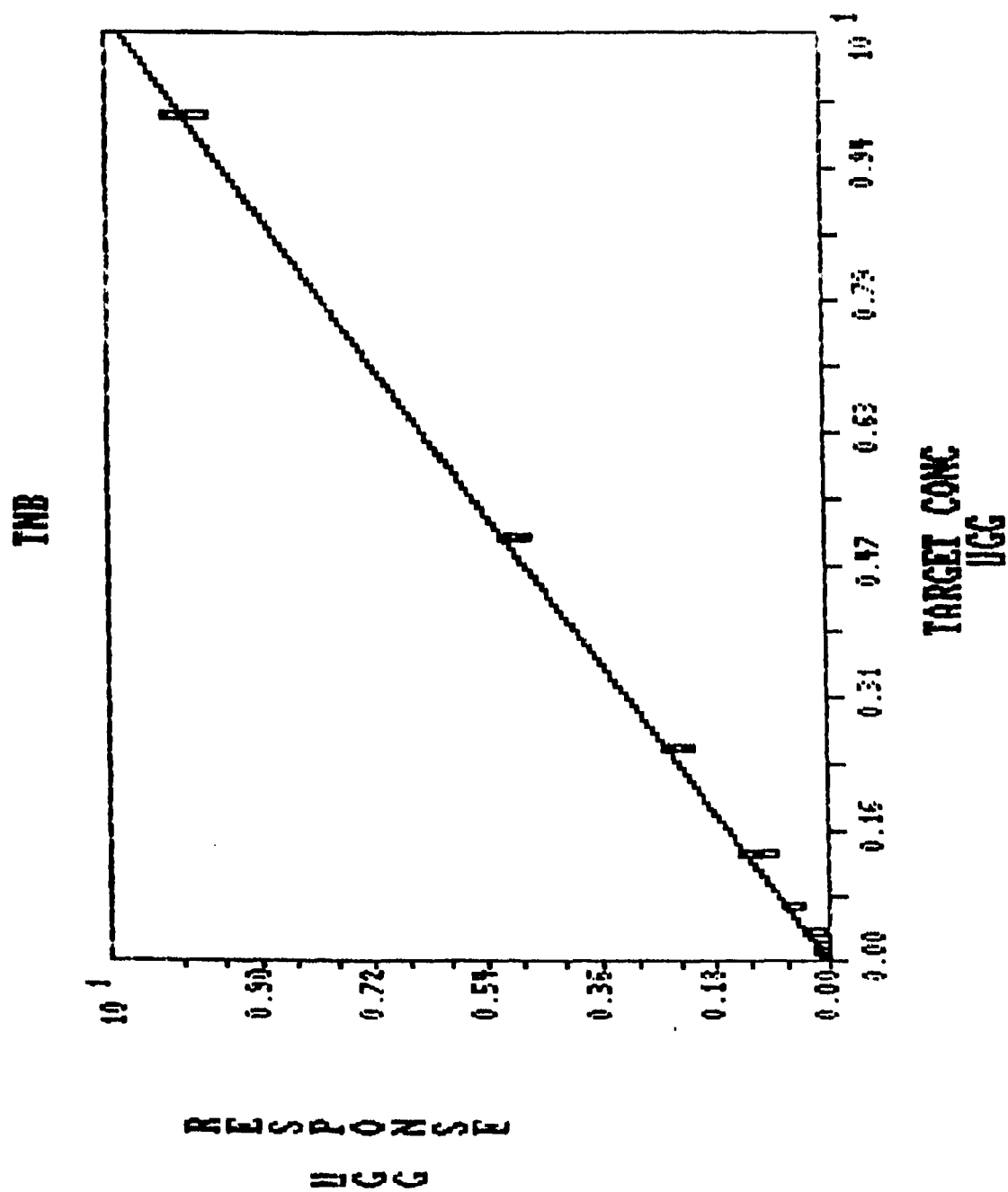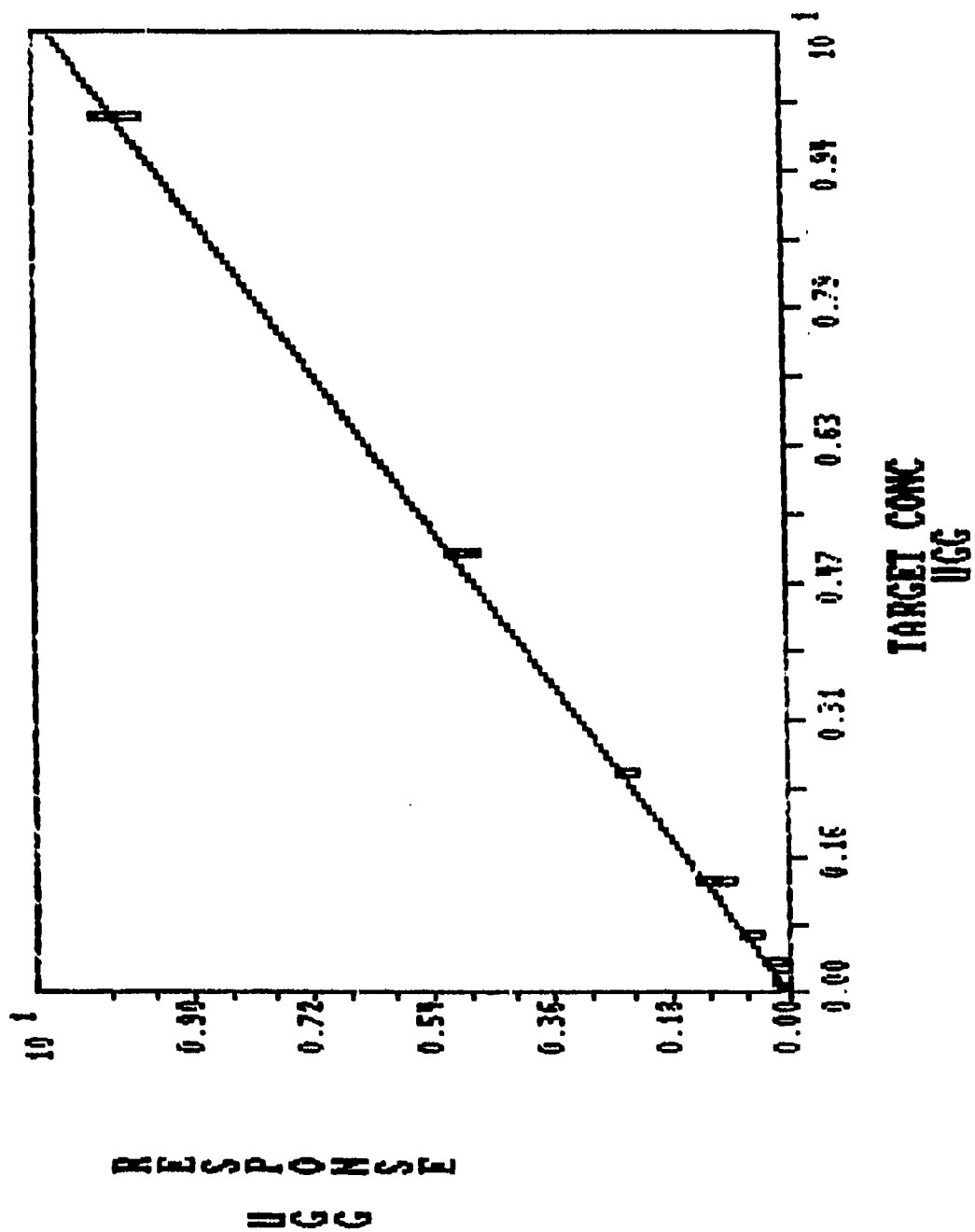
FIGURE F 17a HMX

FIGURE F 17b

HMX

FIGURE F 18a

TNB

RESPONSE
IIGG

TARGET CONC.
IIGG

FIGURE F 18b

FIGURE F 19a

RDX

RESPONSE AREA

TARGET CONC.
ug

FIGURE F 19b

RDX

RESPONSE UGG

TARGET CONC UGG

FIGURE F 20a

TNT

RESPONSE
UGG

TARGET CONC
UGG
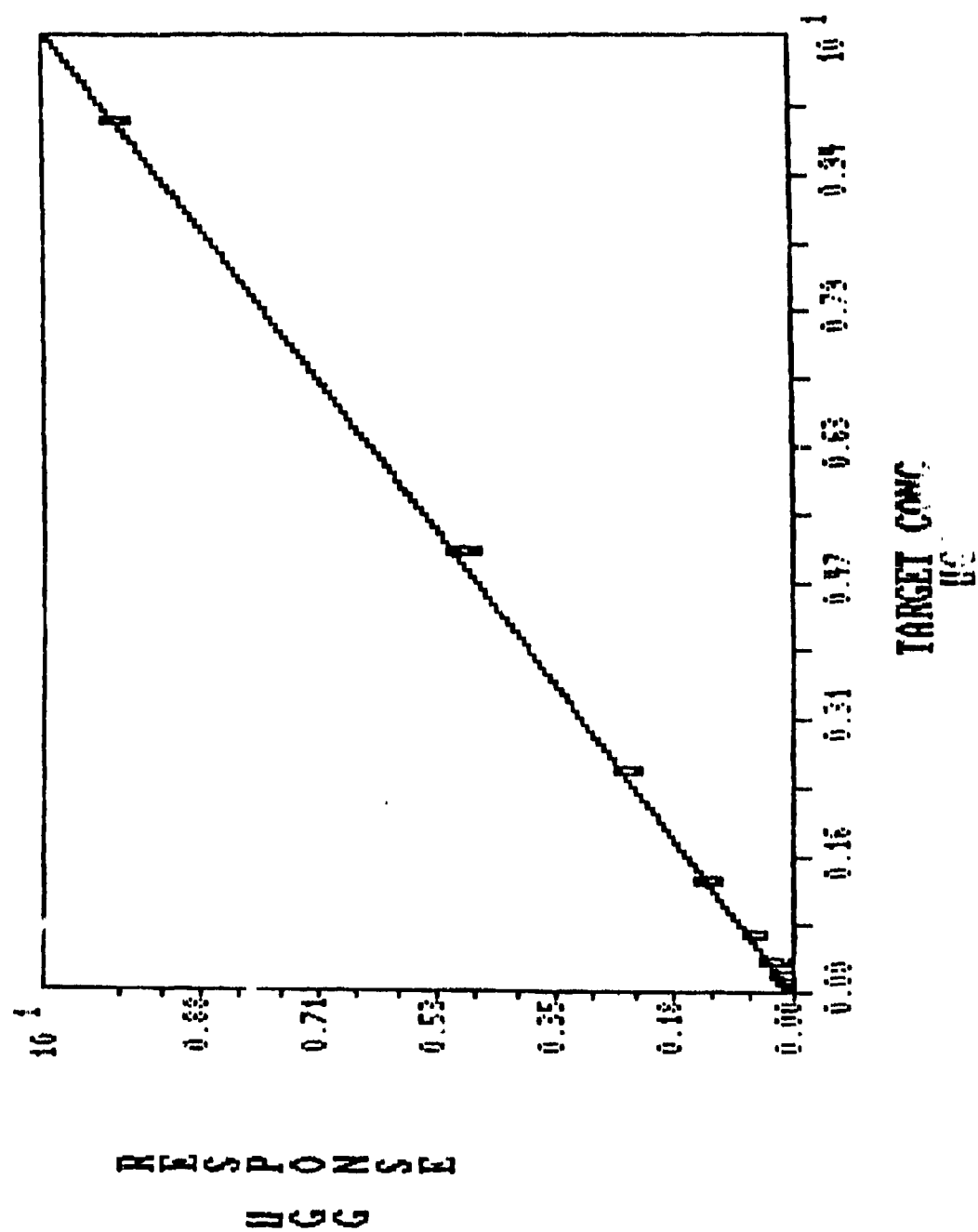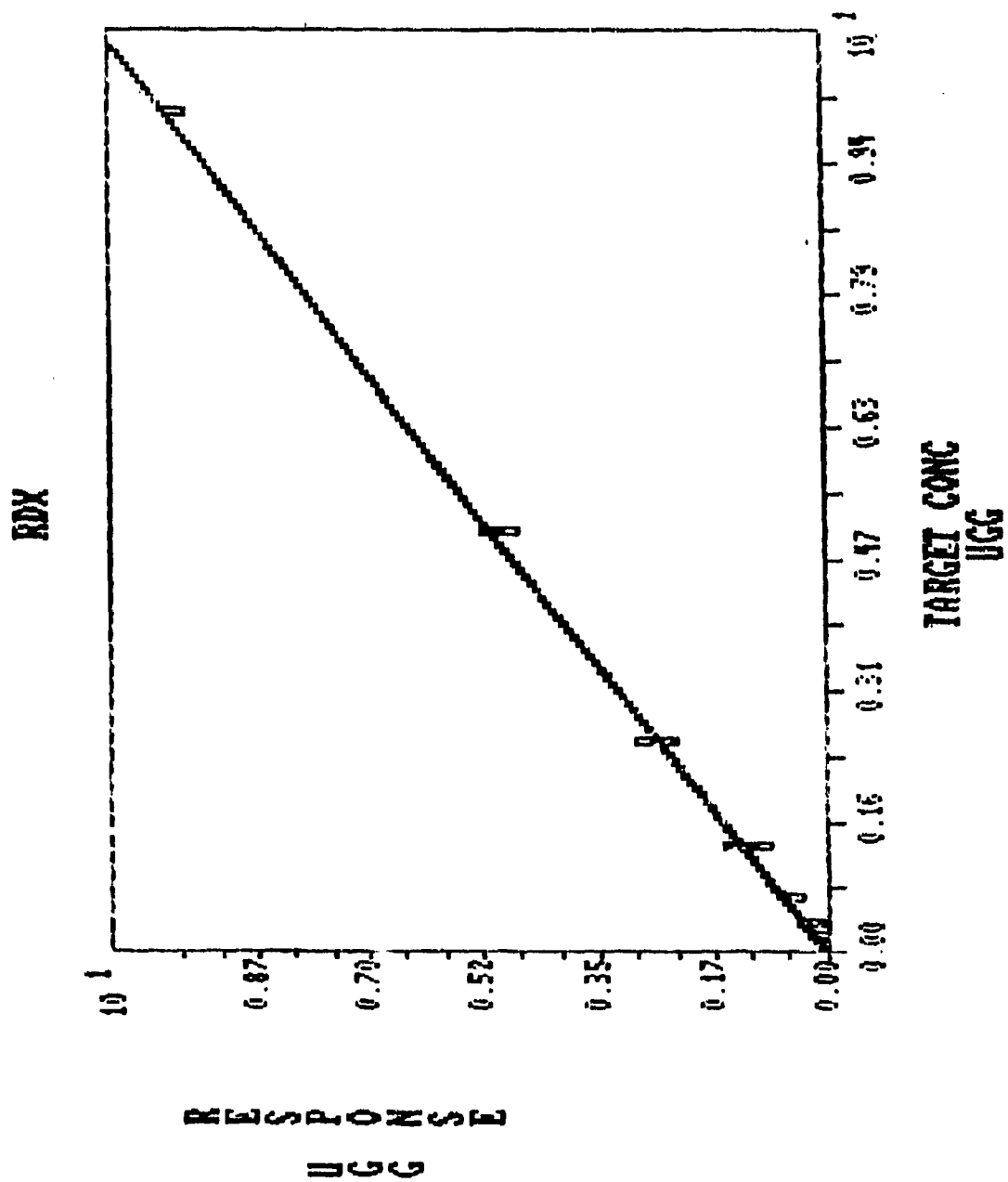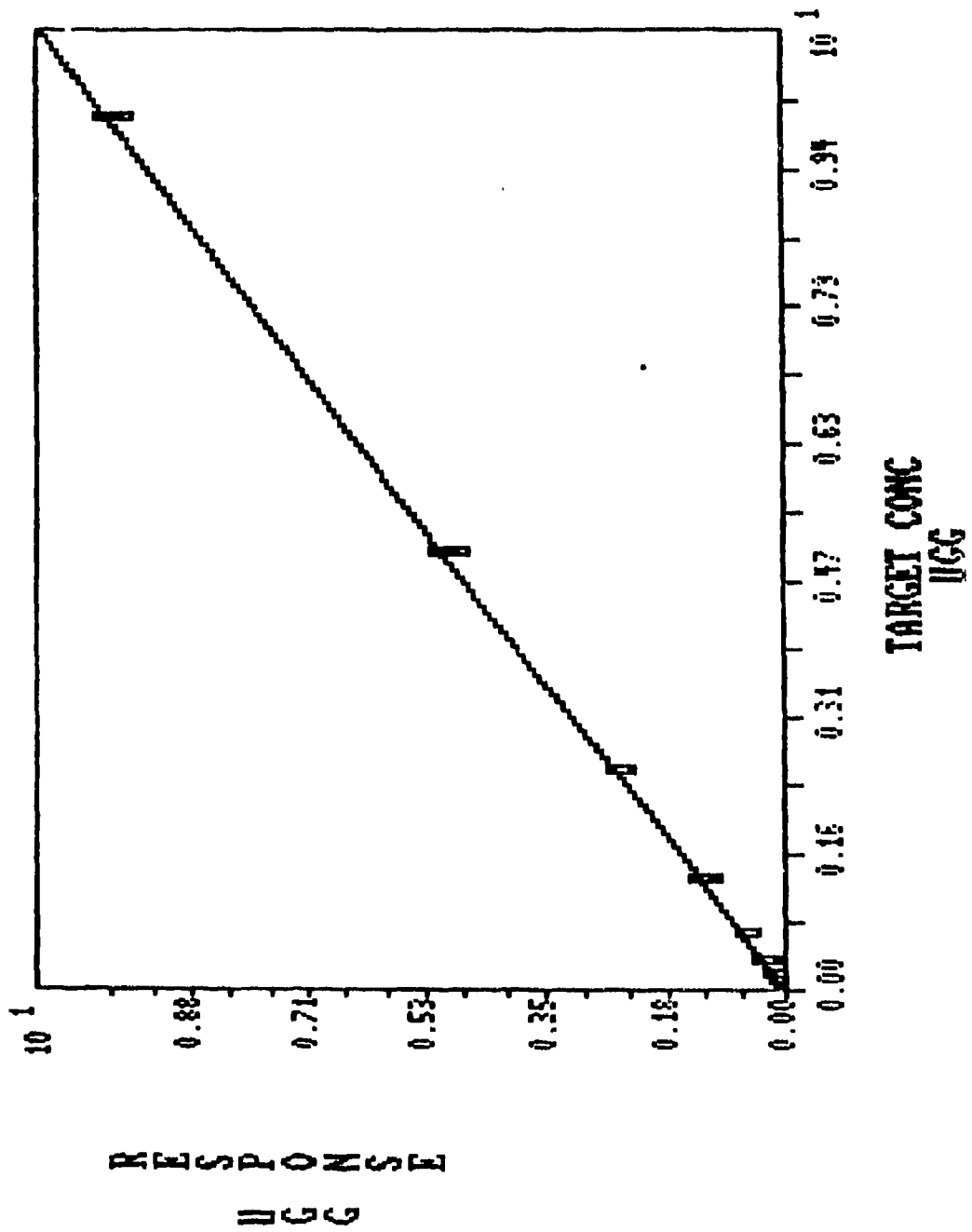
FIGURE F 20b

TNT

FIGURE F 21a

2,4
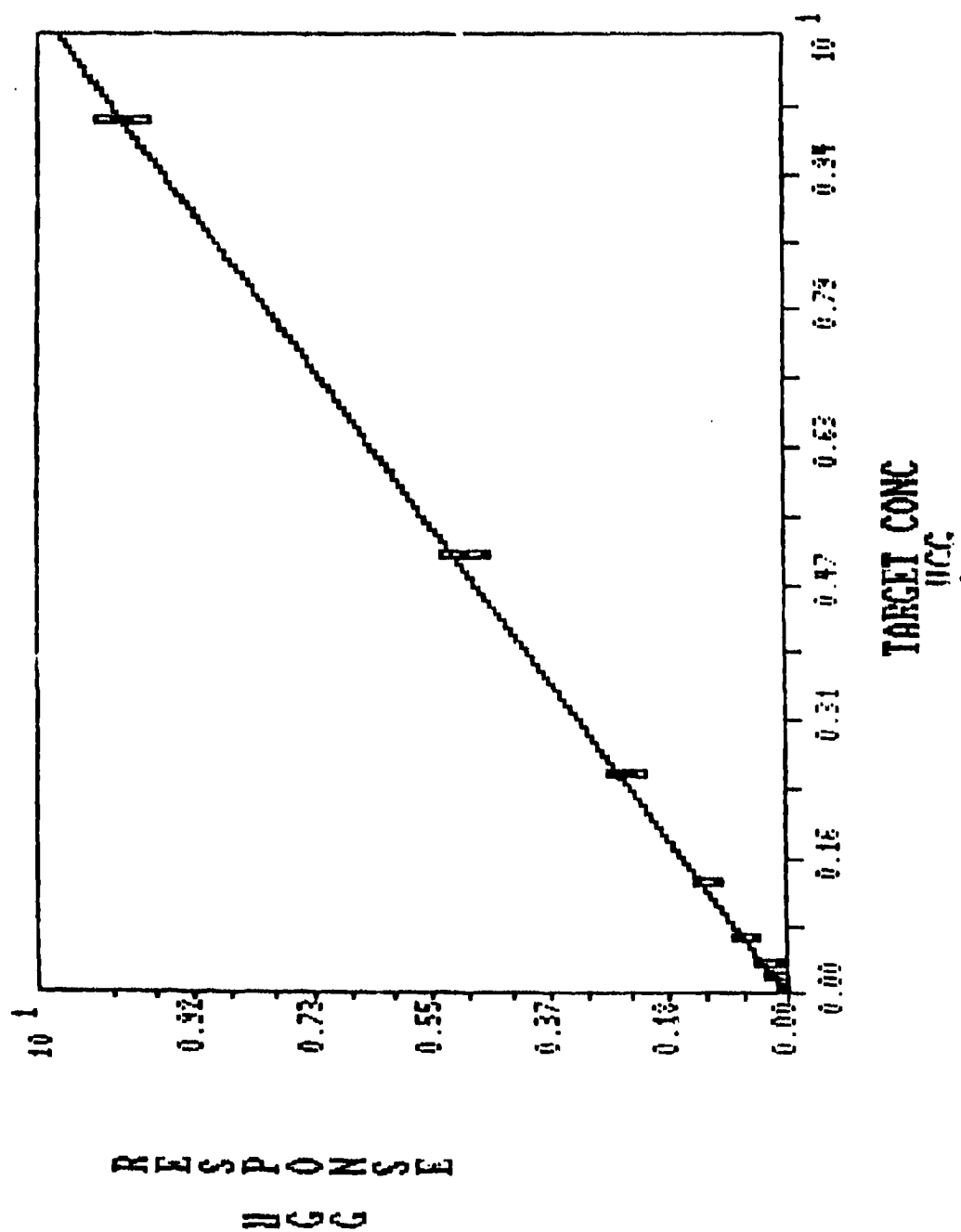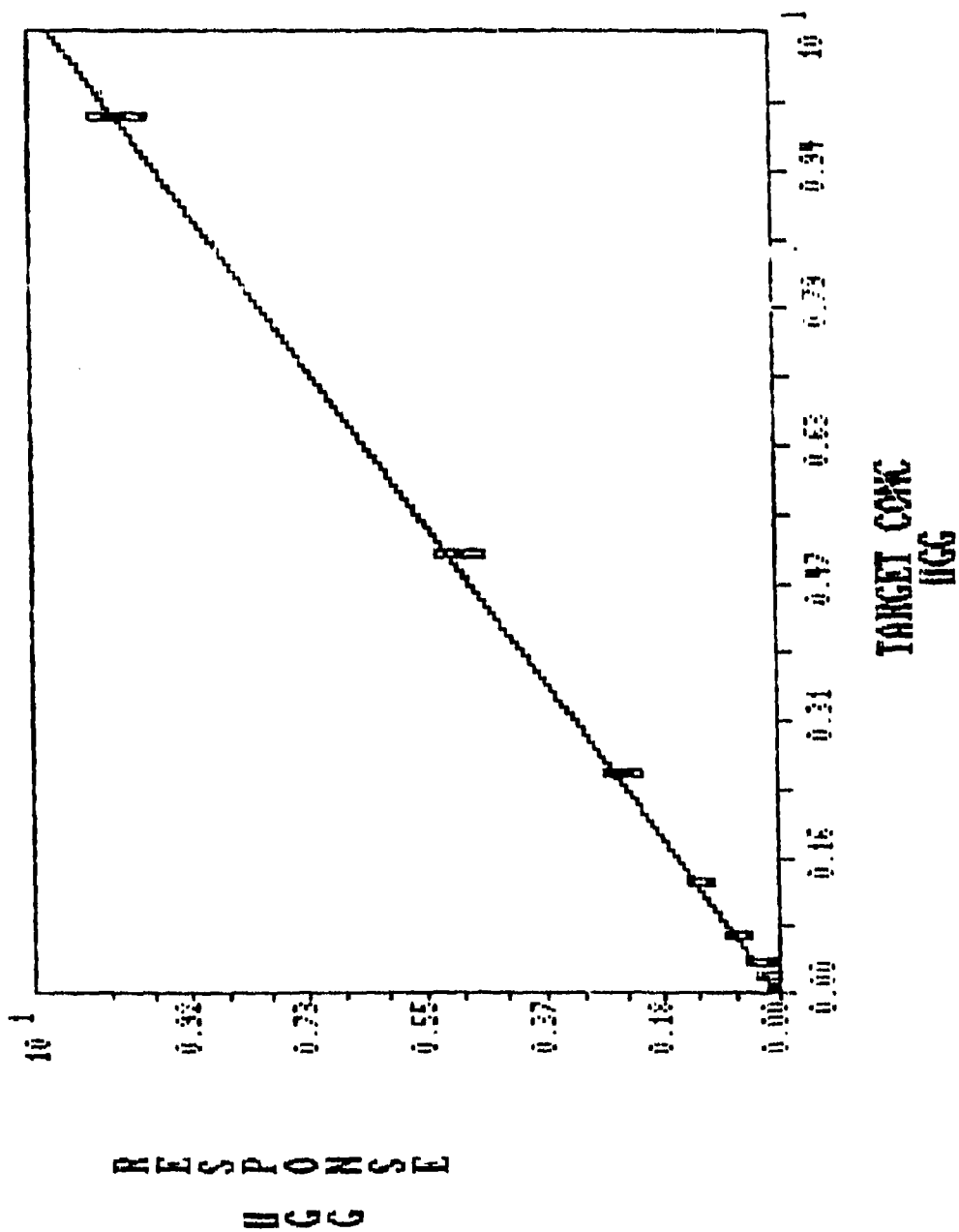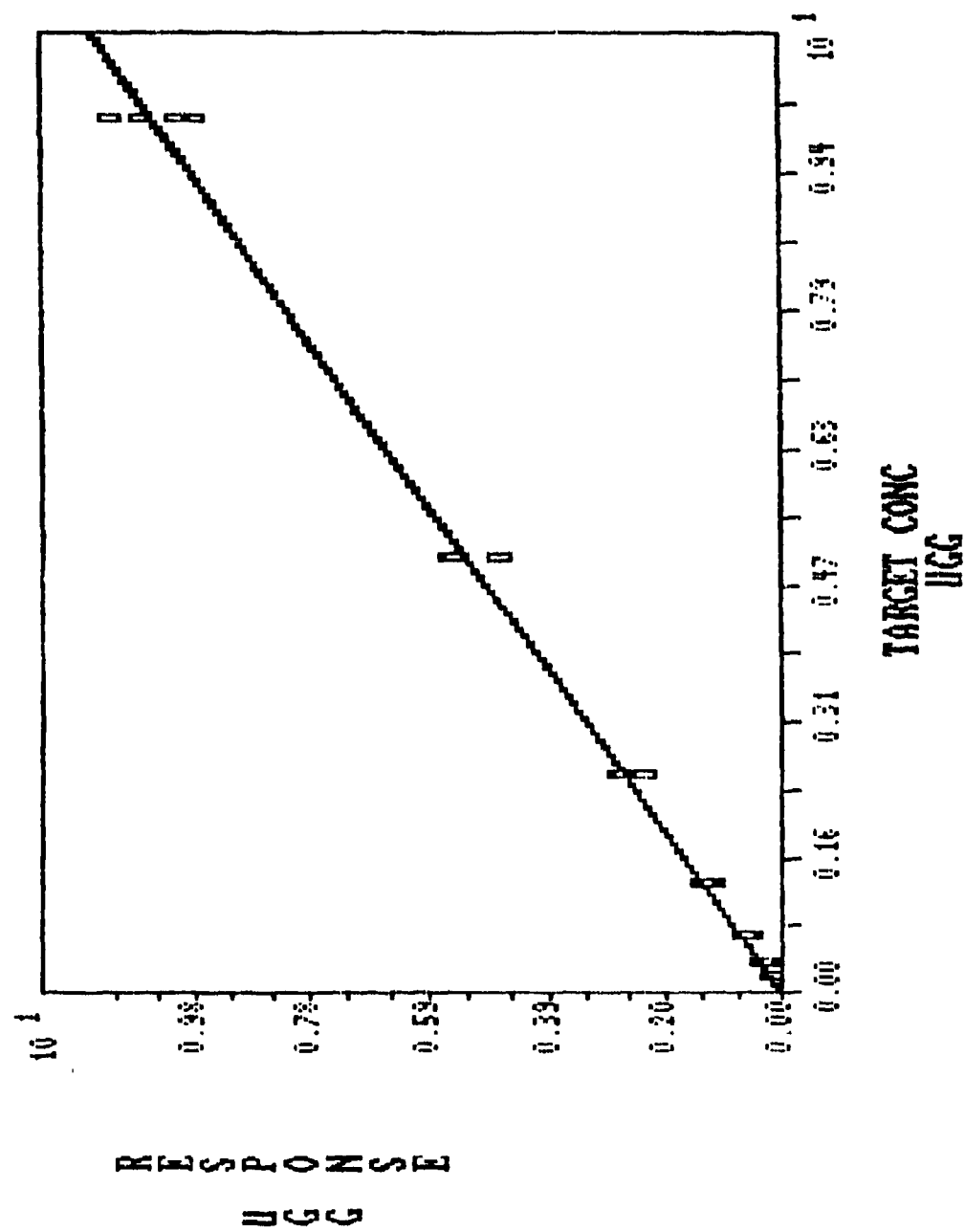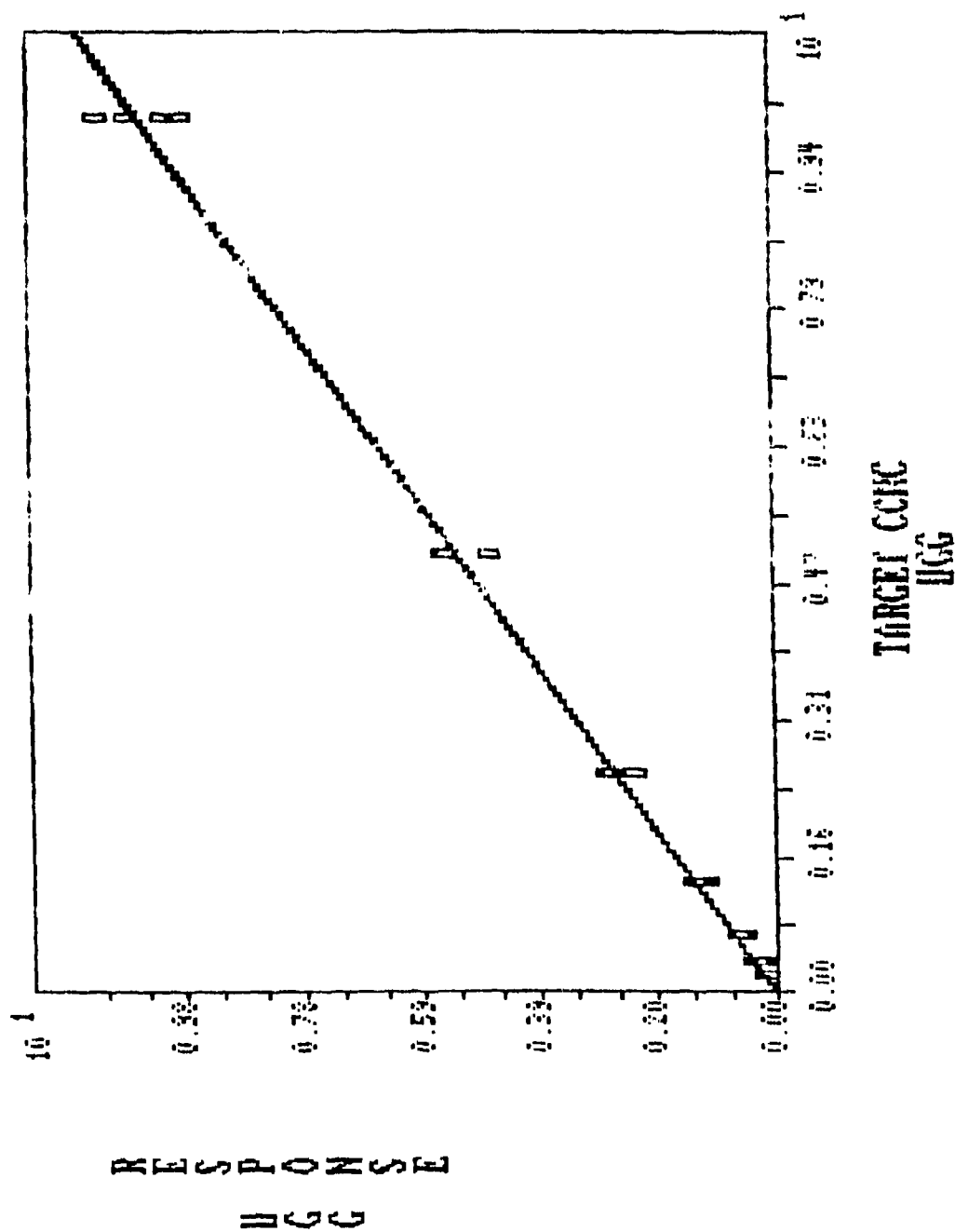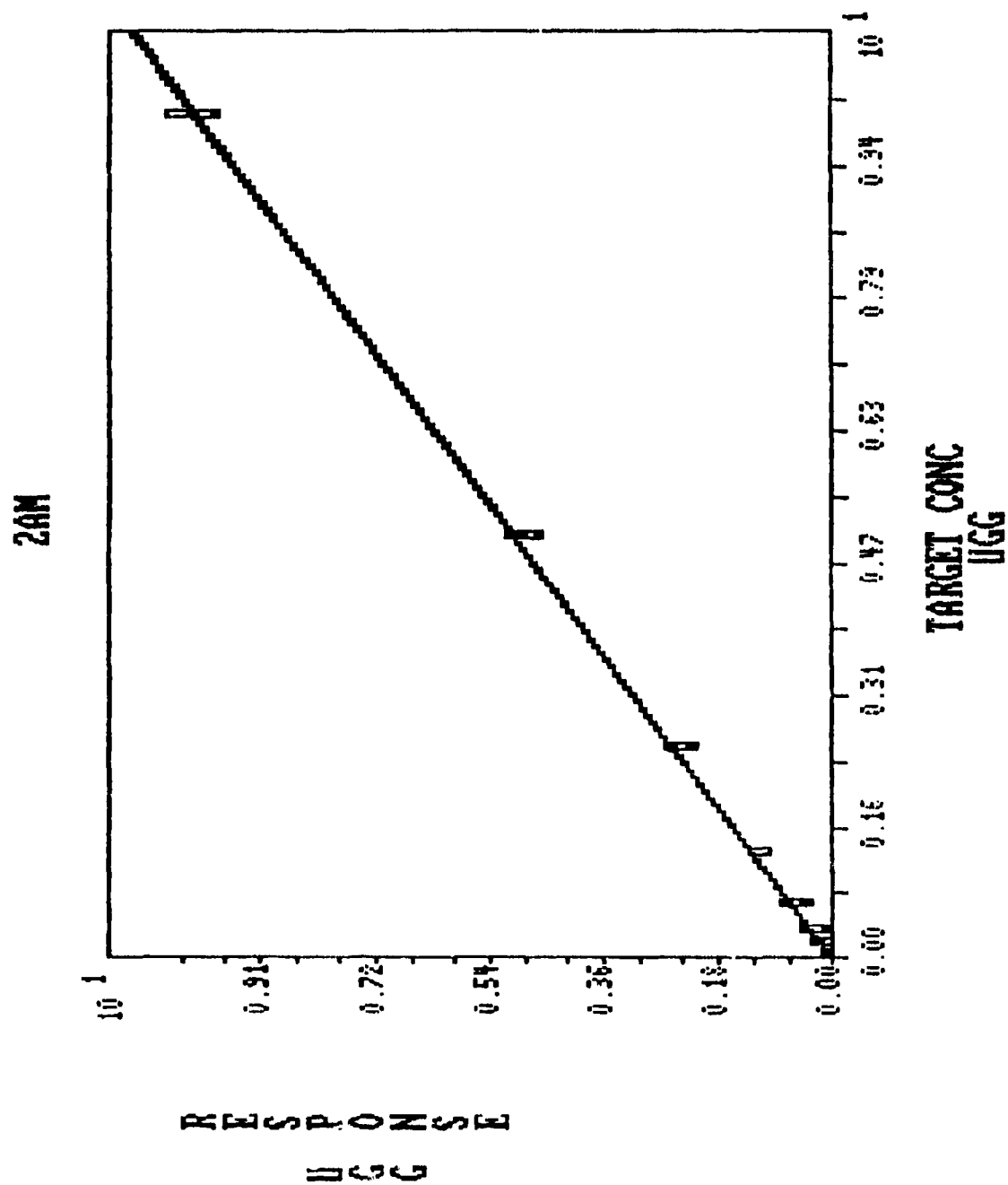
RESPONSE IGG

TARGET CONC
UG.

FIGURE F 21b

2-4DNT

RESPONSE
HGG

TARGET CONC
HGG

FIGURE F 22a

2,6

RESPONSE UGG

TARGET CONC UGG

FIGURE F 22b

2-6DNT

RESPONSE
UG/G

TARGET CONC
UG/G

FIGURE F 23a

2AM

RESPONSE UGG

TARGET CONC
UGG

FIGURE F 23b

SUMMARY

RESPONSE =IGG

TARGET CONC. HGG

FIGURE F 24a

4AMDNT

RESPONSE
UGG

TARGET CONC
UGG

FIGURE F 24b

4AMDNT

RESPONSE
UGG

TARGET CONC
UGG

Figure F 25a

HMX

FOUND CONC UGG

TARGET CONC UGG

FIGURE F 25b

HMX

TARGET CONC
UGG

FOUND CONC
UGG

FIGURE F 26a

TMB

FOUND CONC
UGG

TARGET CONC
UGG

FIGURE F 26b

TNB

FOUND CONC UG

TARGET CONC UGG

FIGURE F 27a

RDX

FOUND CONC
UGG

TARGET CONC
UGG

FIGURE F 27b

RDX

TARGET CONC
UGG

FOUND CONC
UGG

FIGURE F 28a

TNT

TARGET CONC
UGG

FOUND CONC
UGG

FIGURE F 28b

TNT

TARGET CONC
UGG

FOUND CONC
UGG

FIGURE F 29a

2,4

TARGET CONC
UGG

FOUND CONC UGG

FIGURE F 29b

2-4DNT

TARGET CONC
UGG

FOUND CONC
UGG
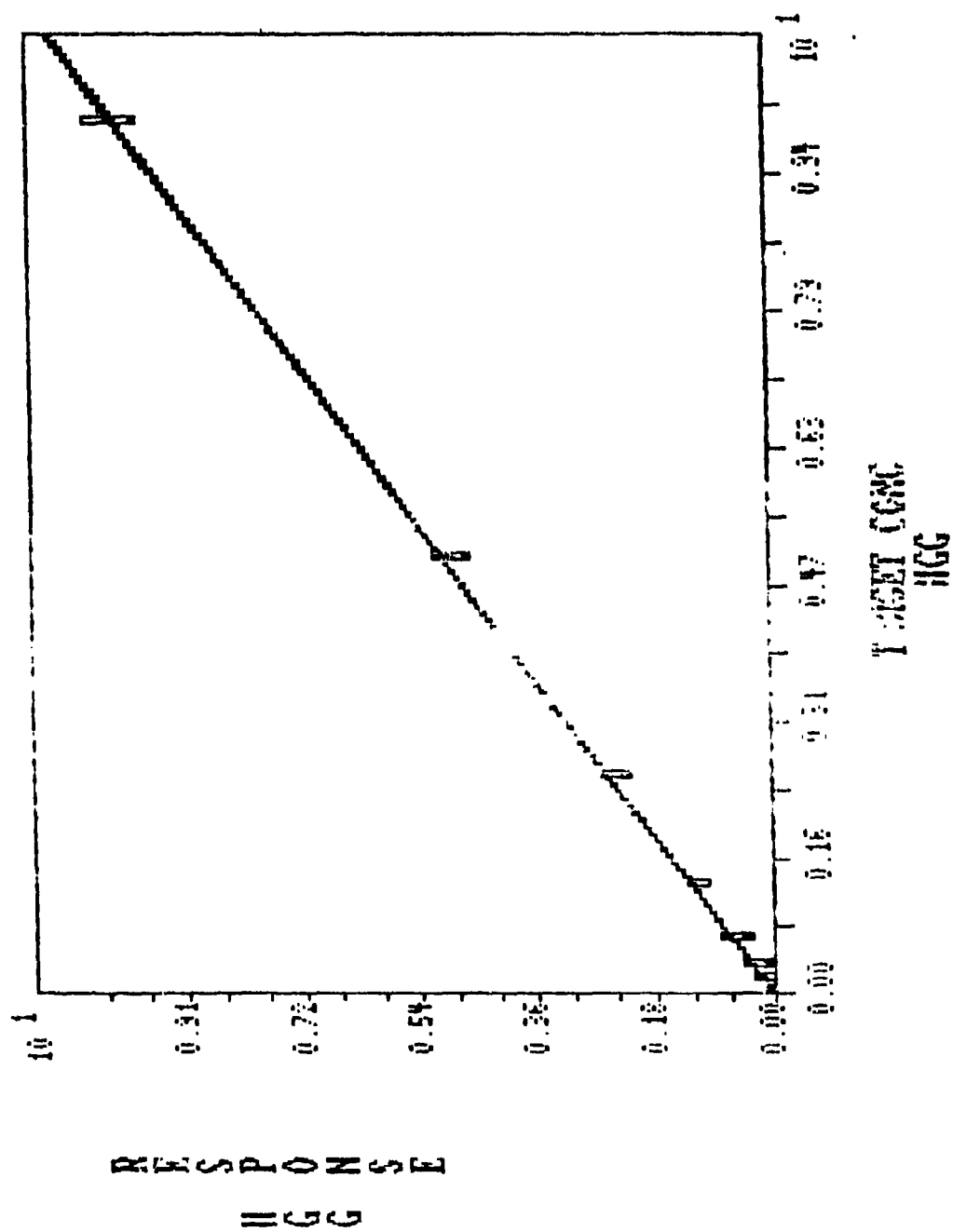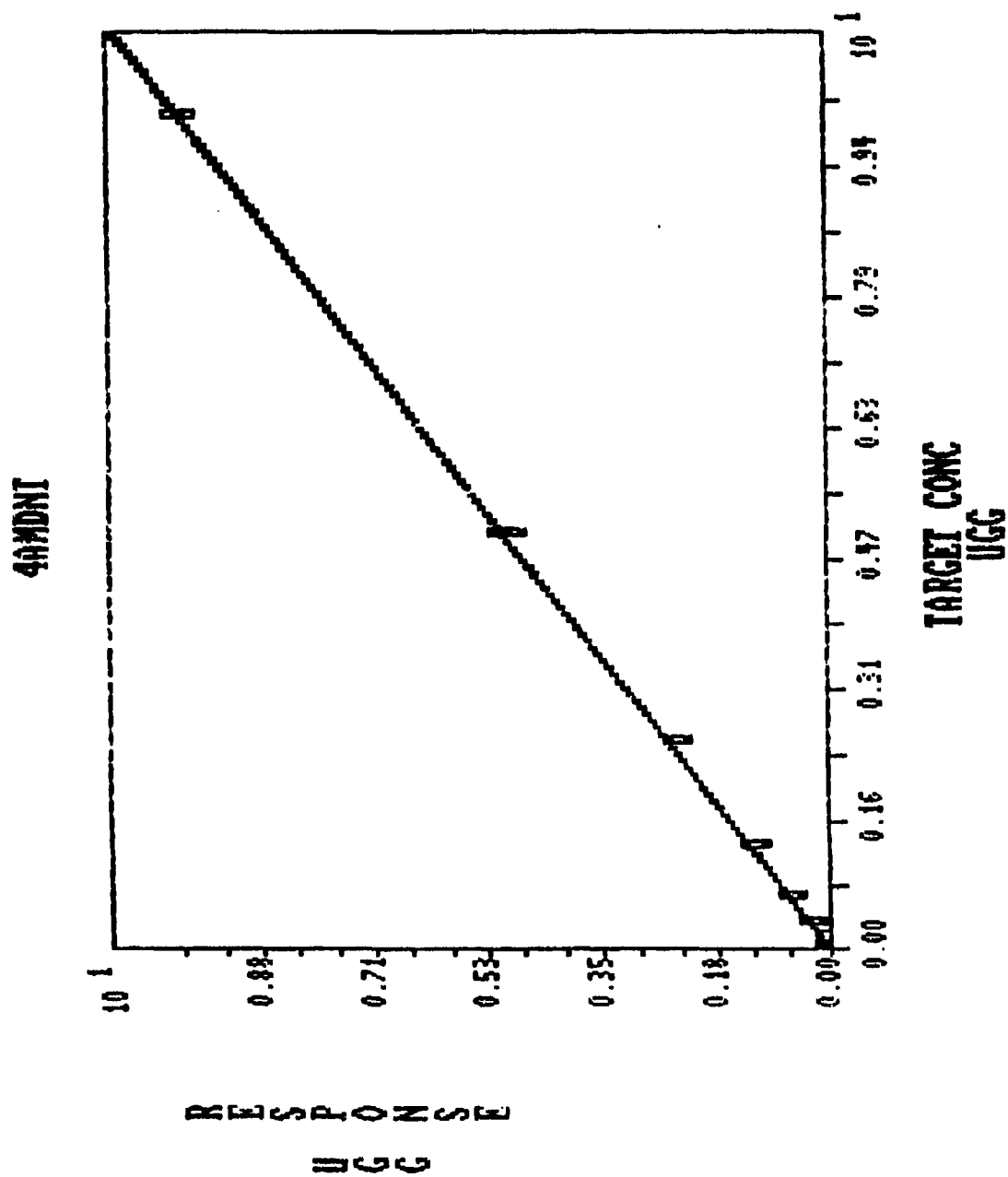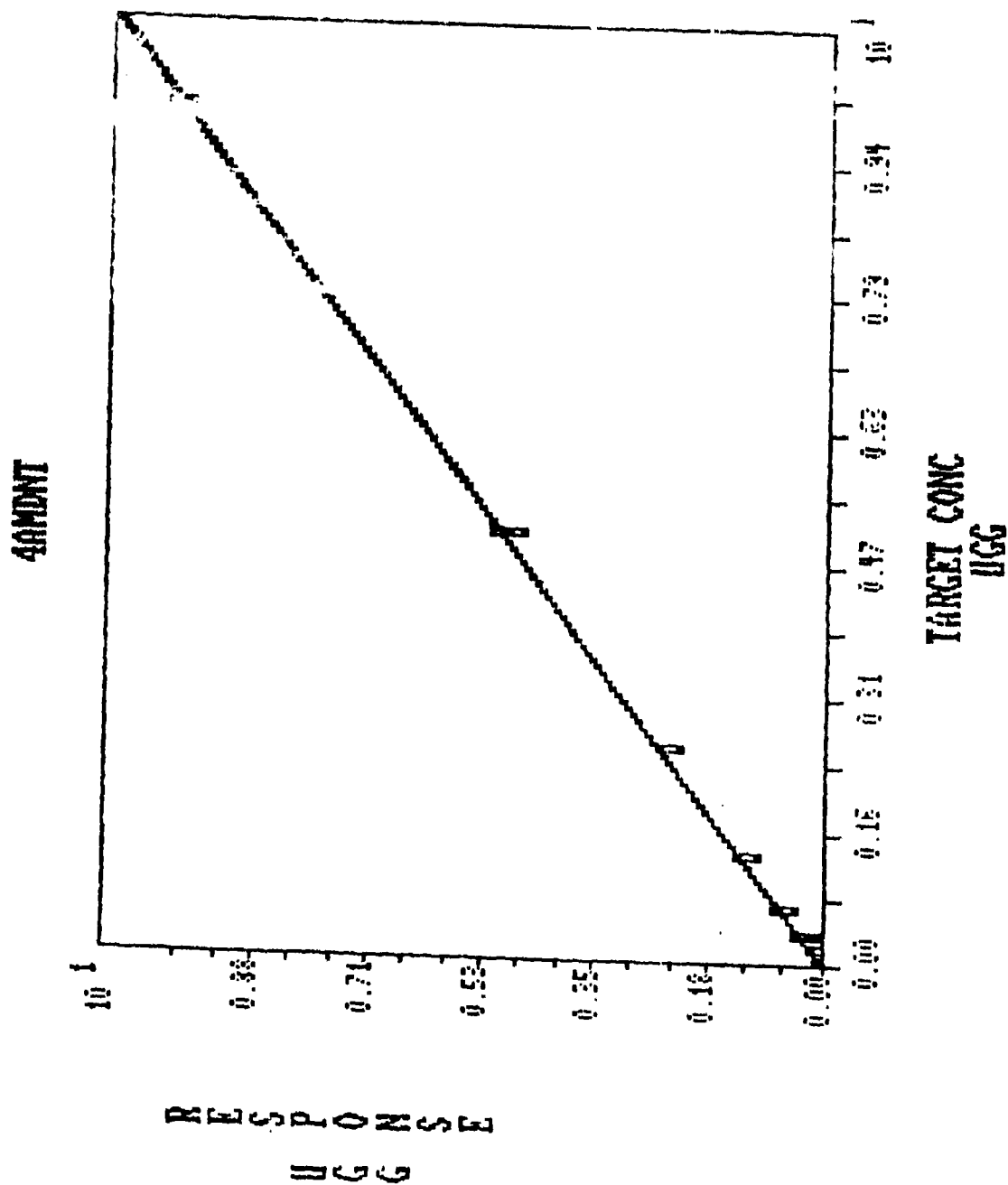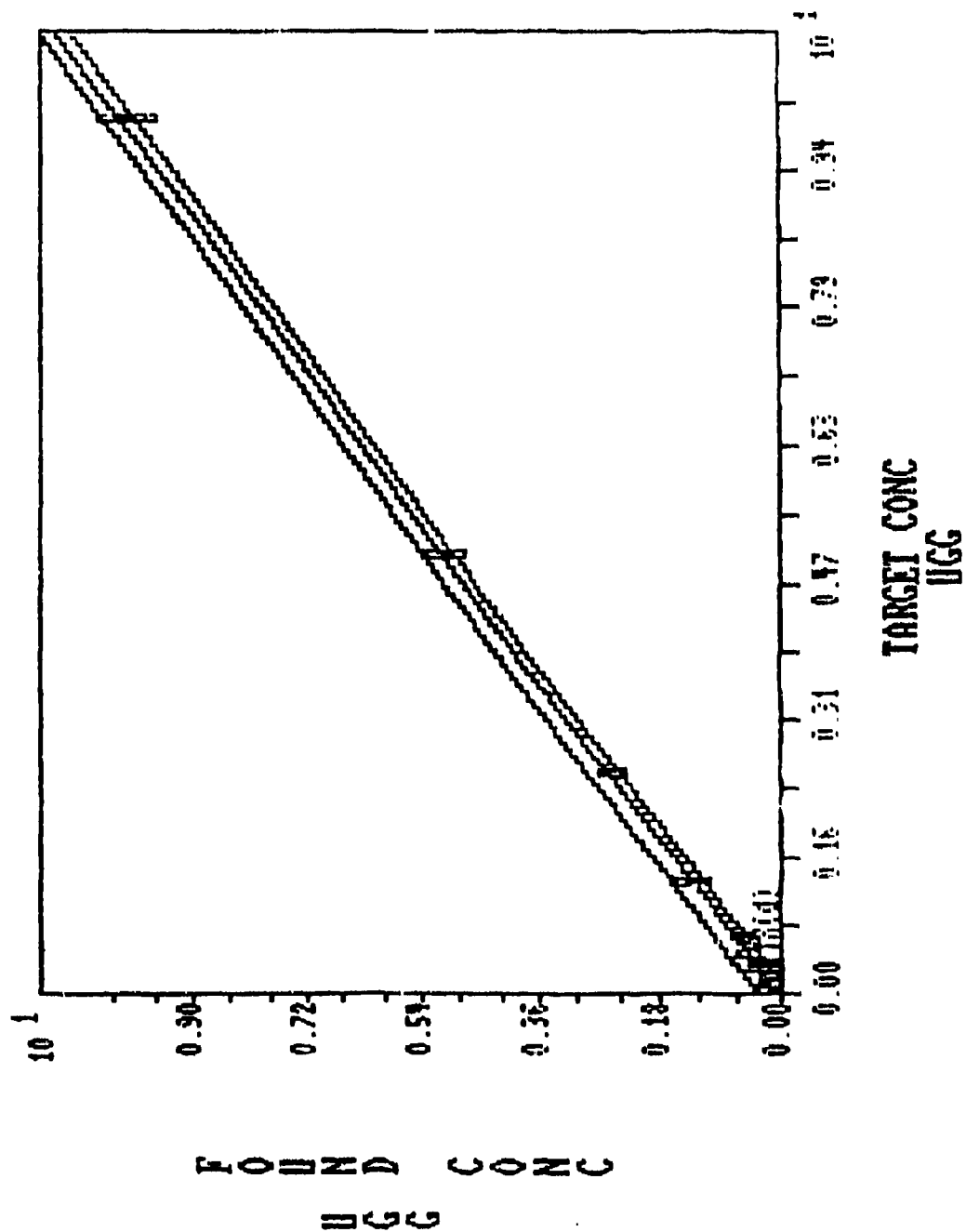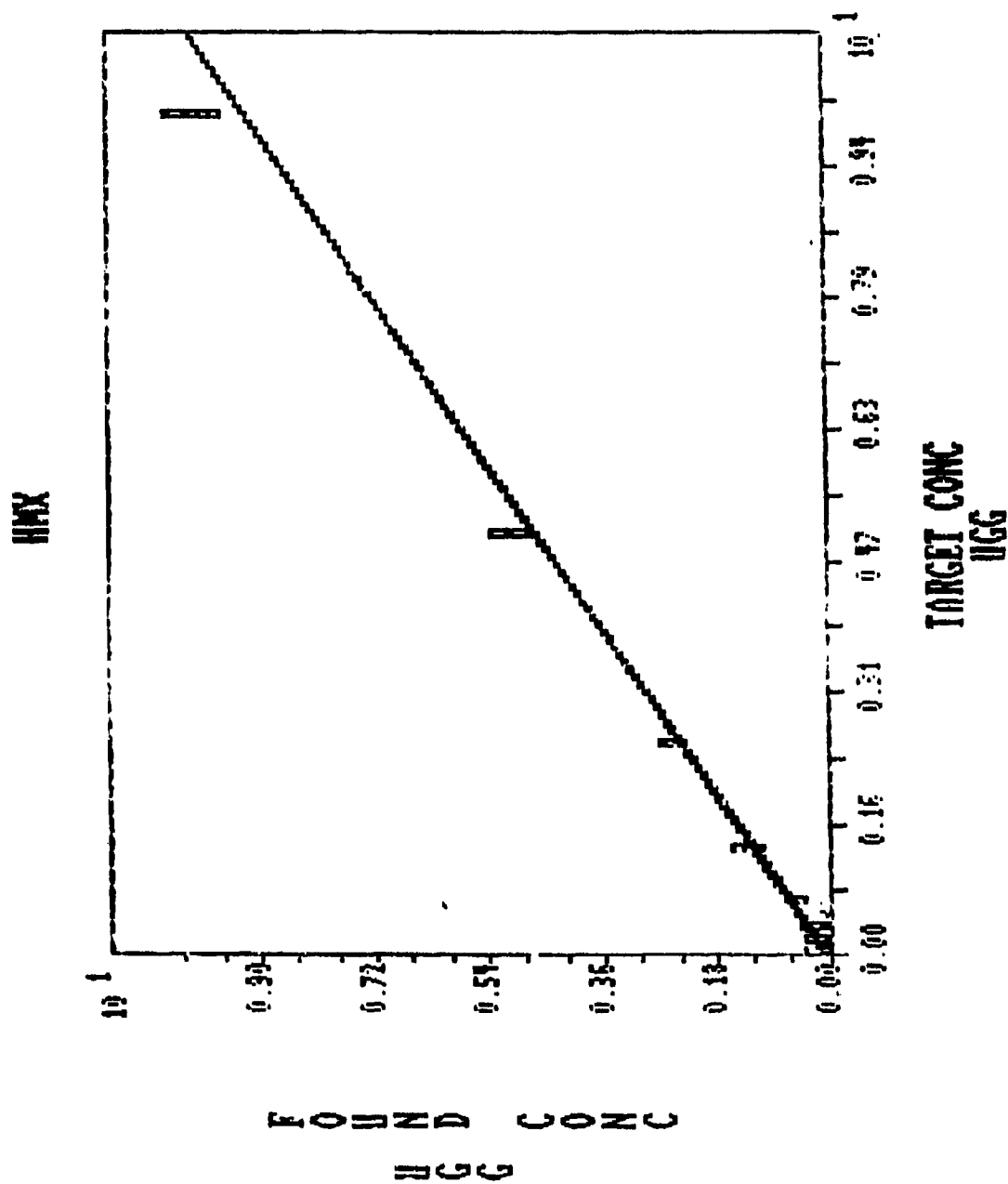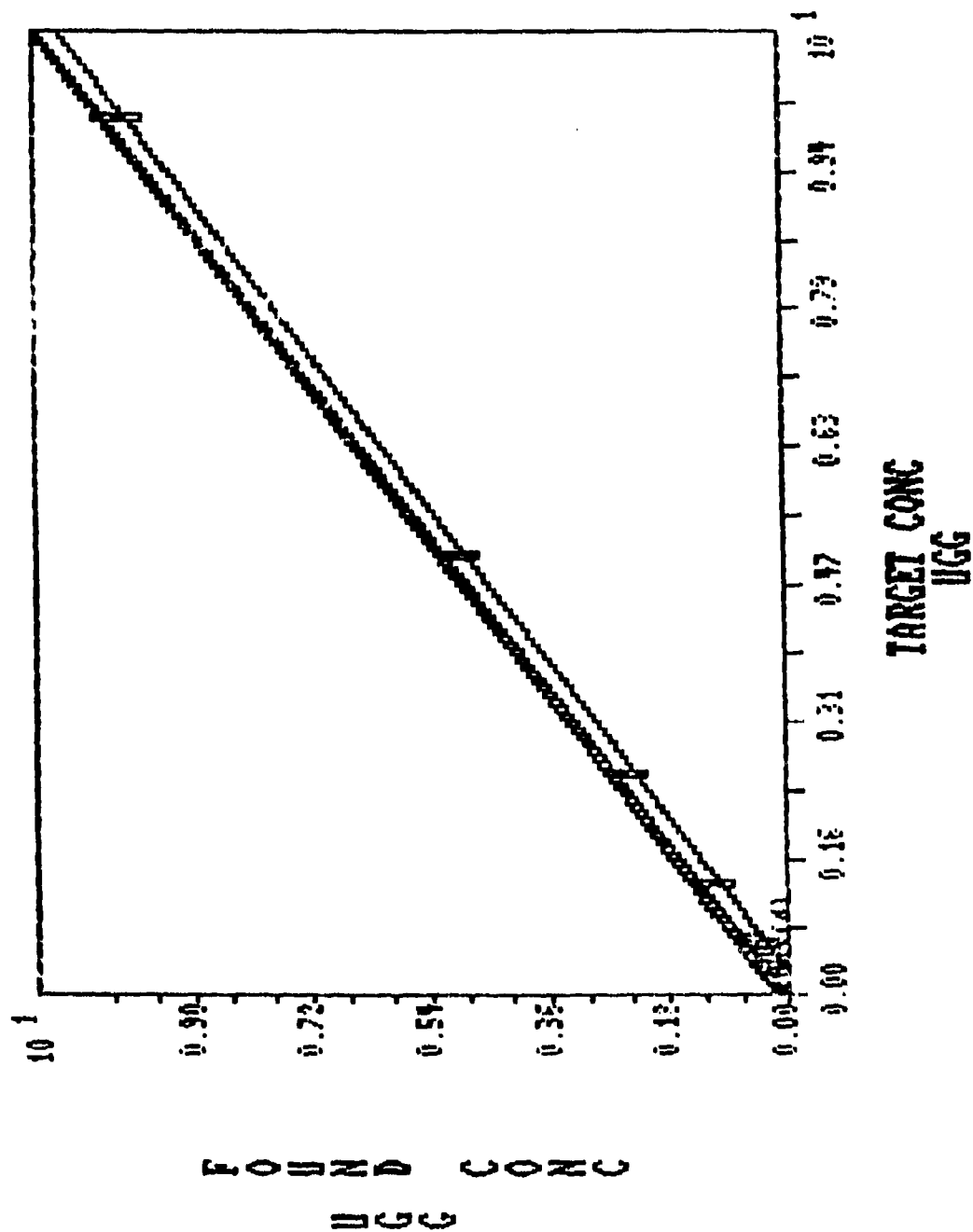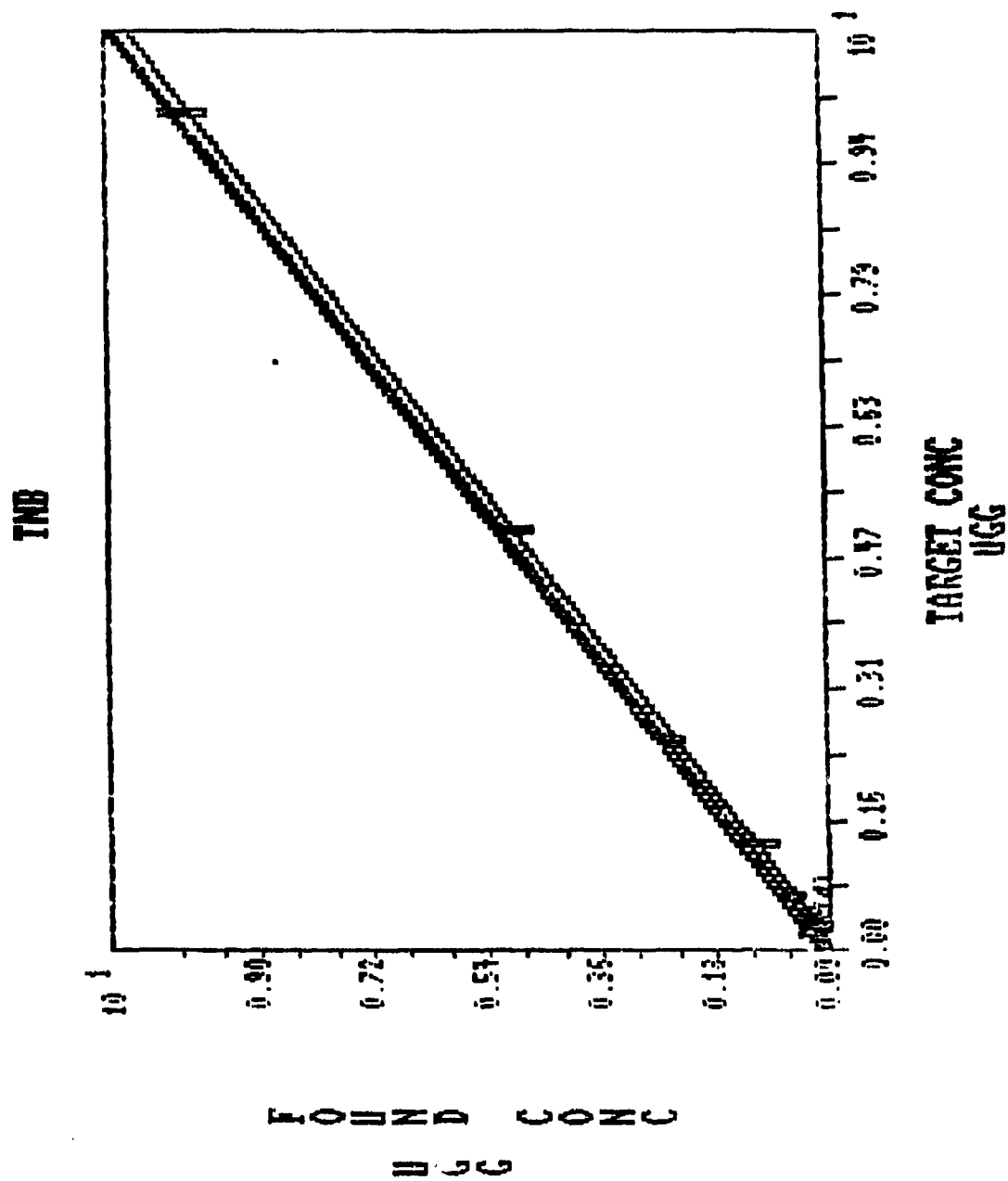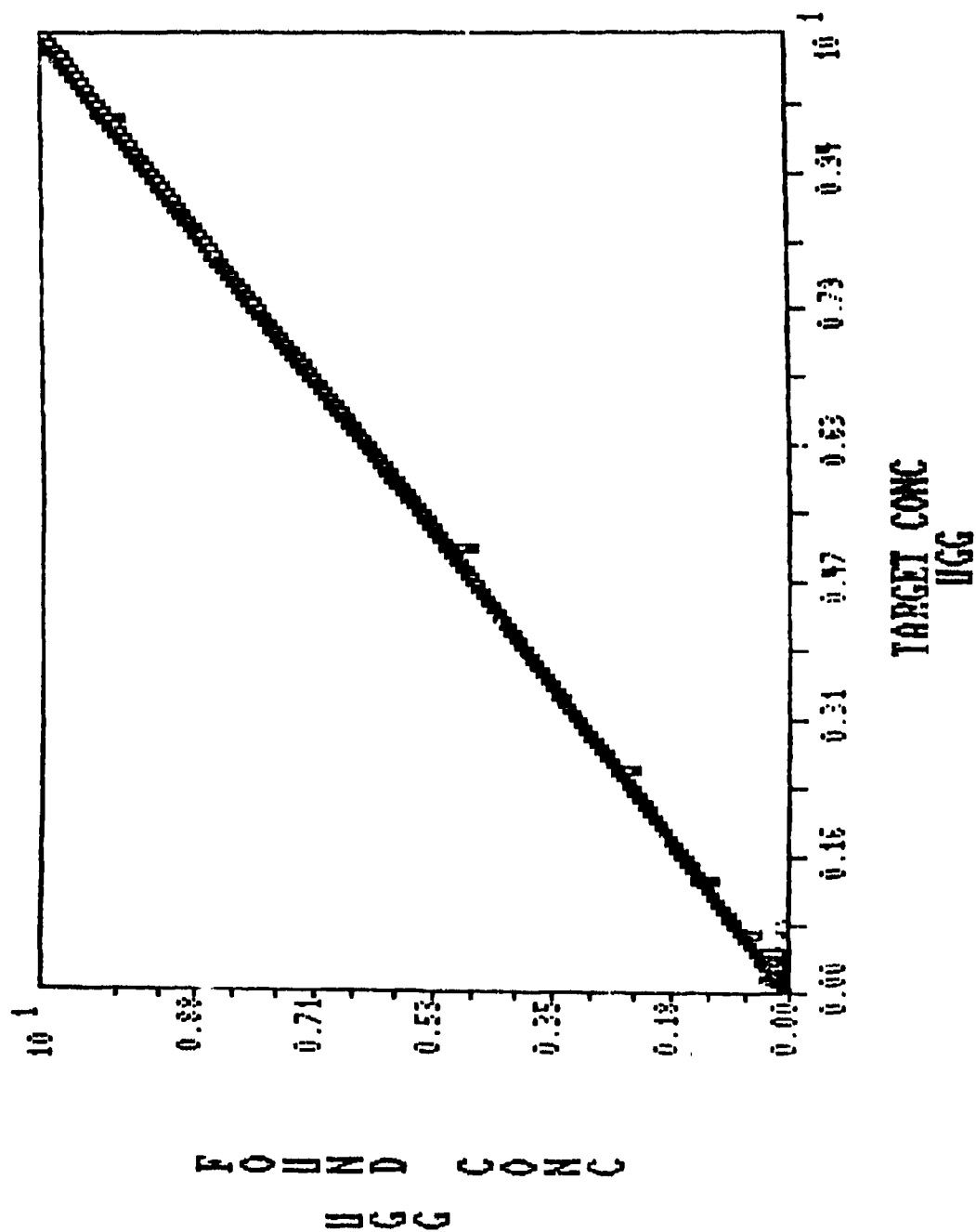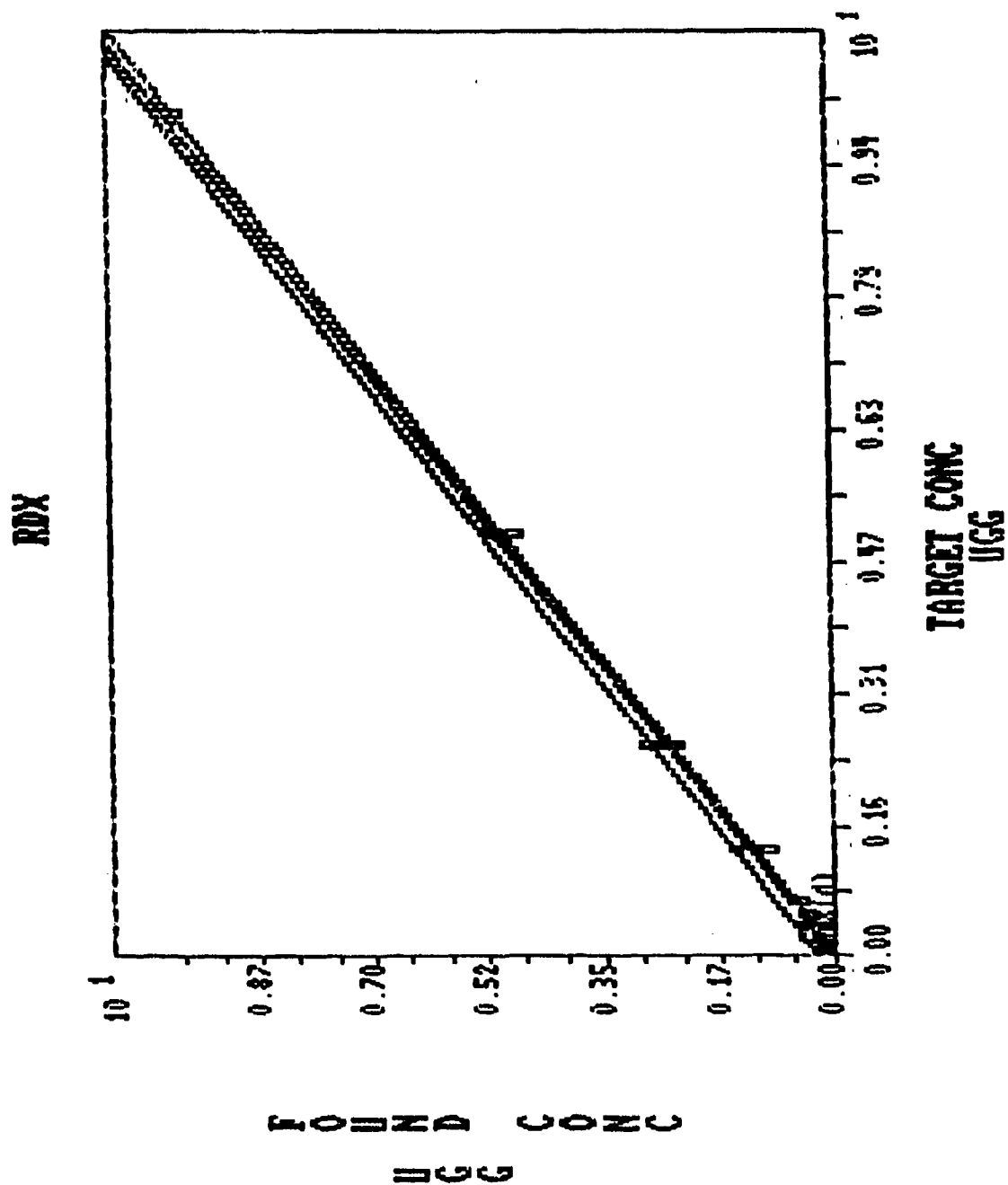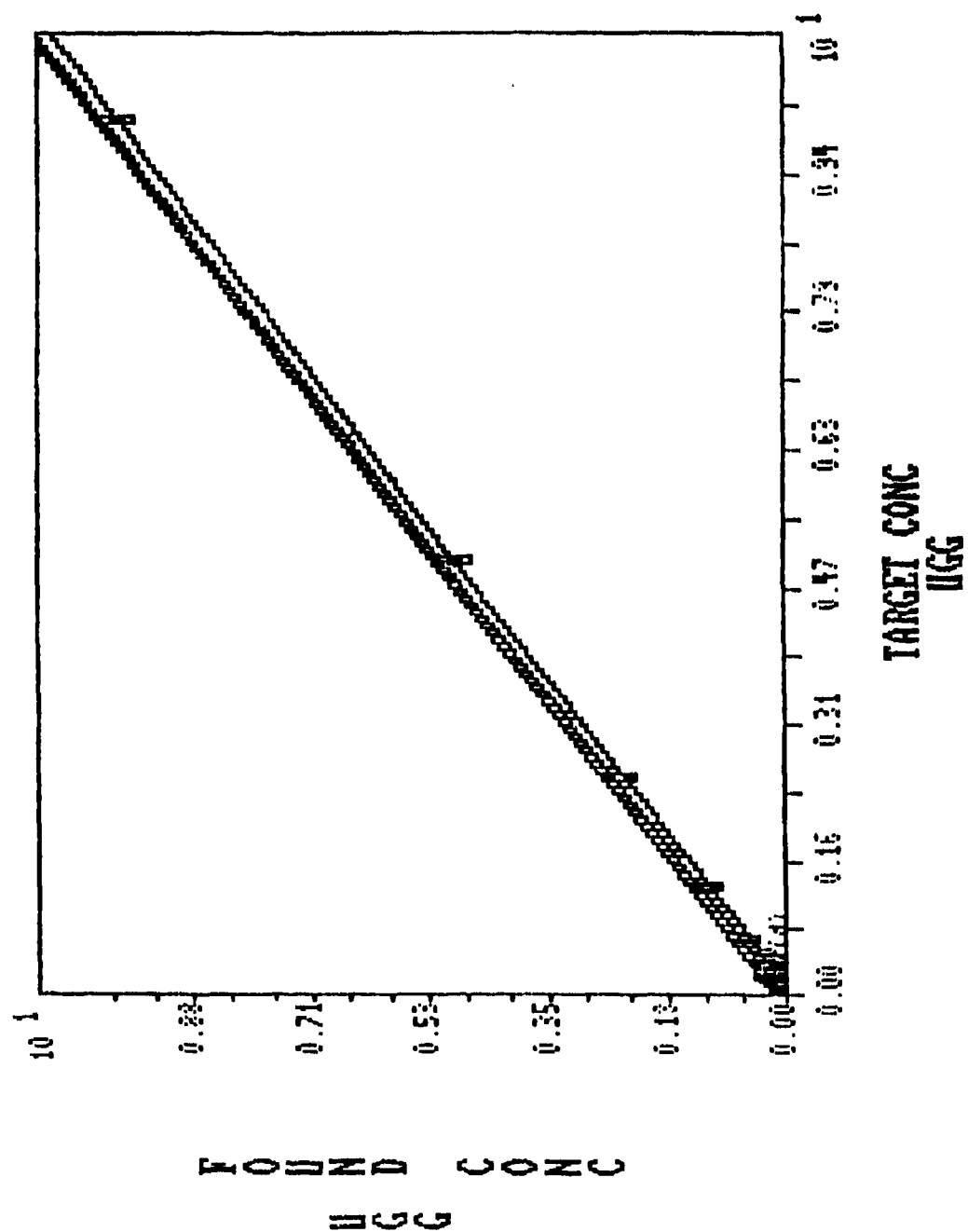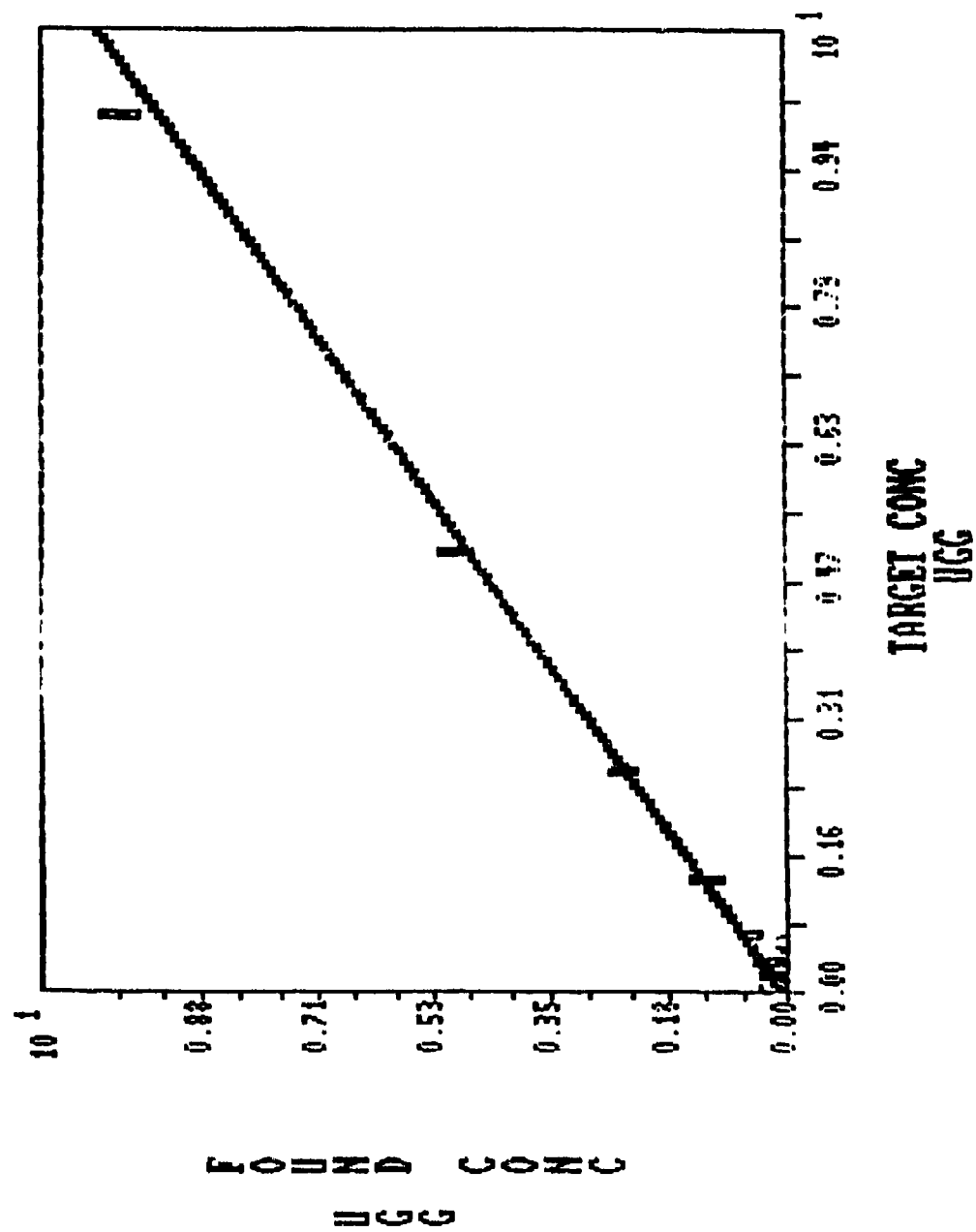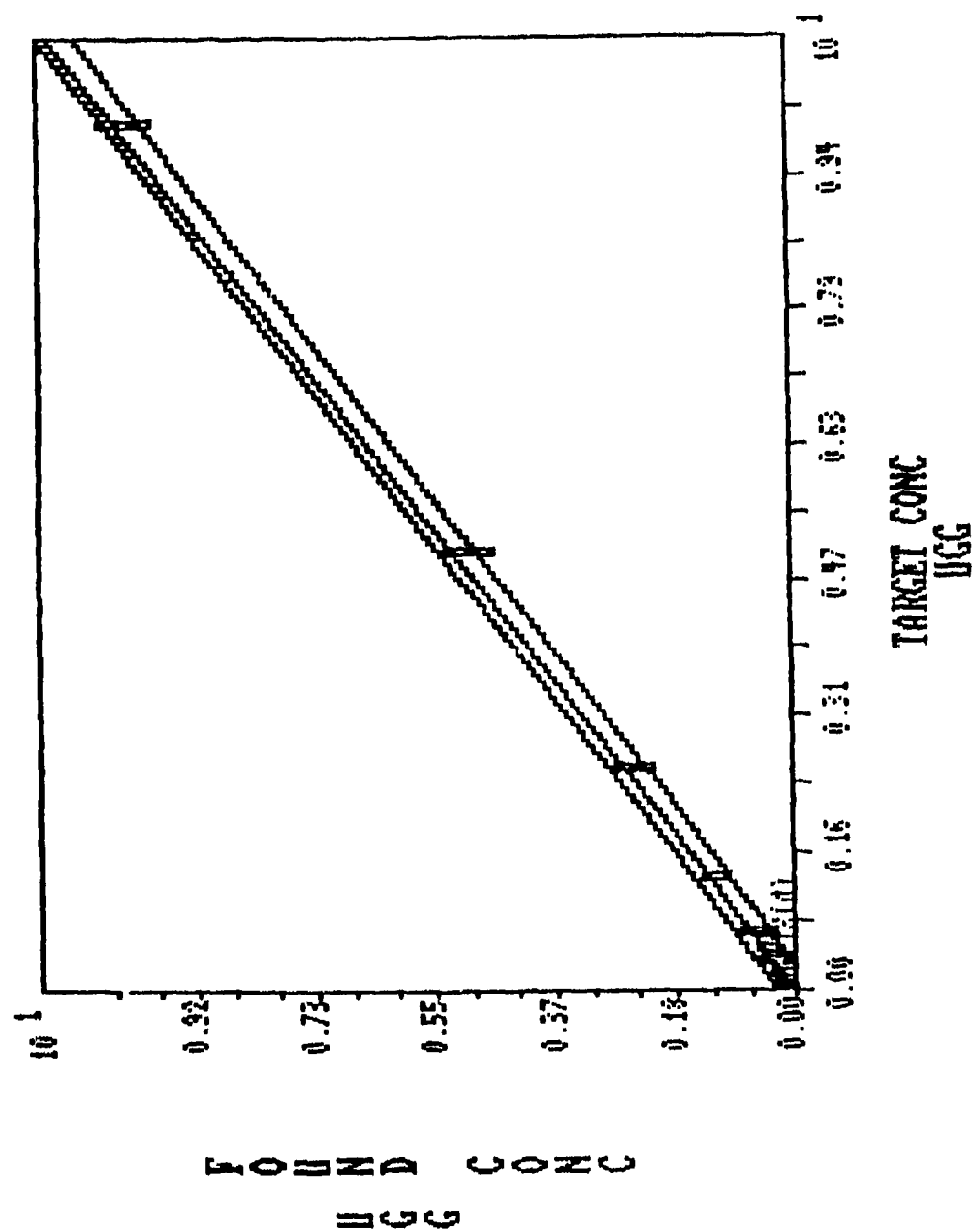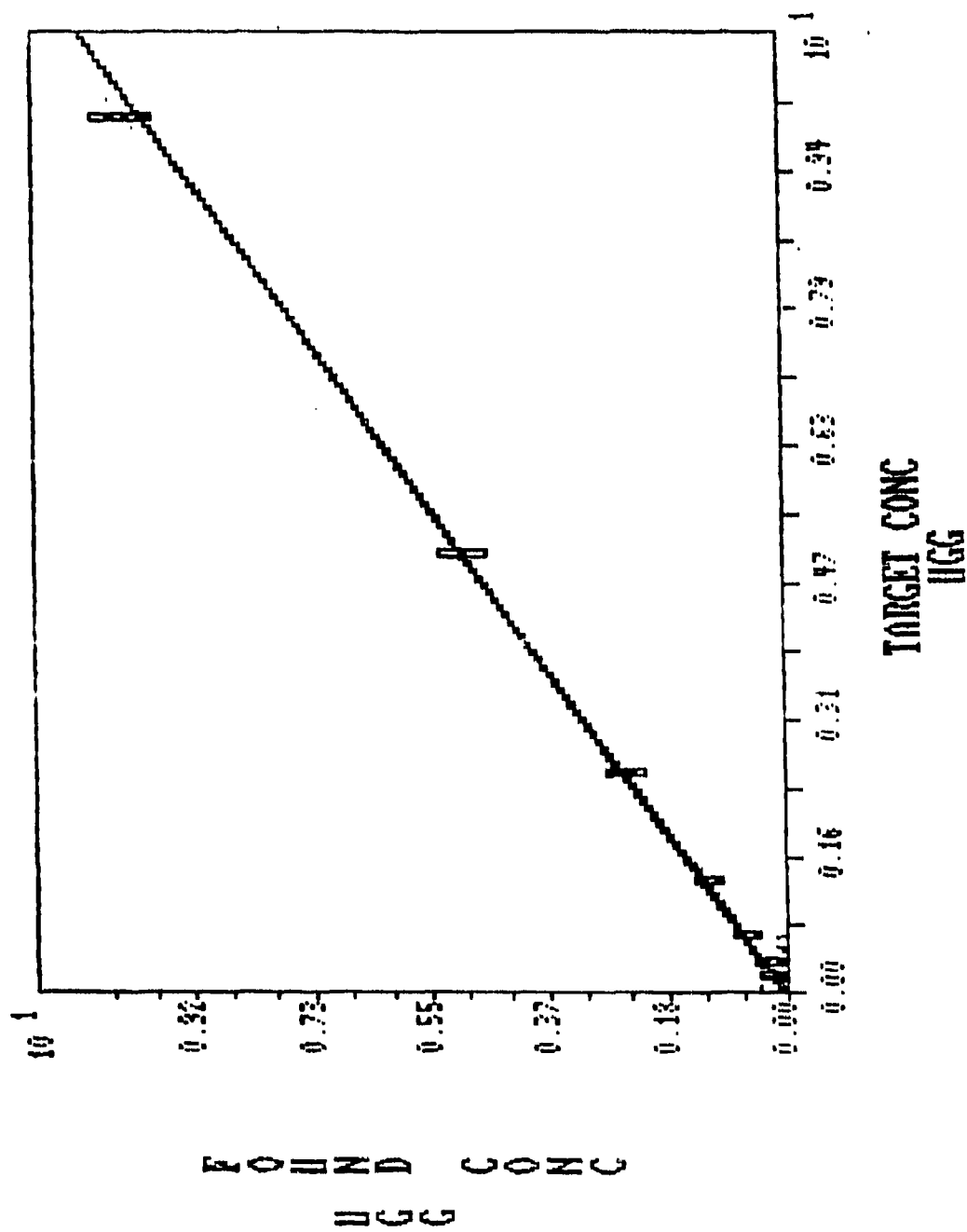
FIGURE F 30a

2,6

FIGURE F 30b

2-6DNT

TARGET CONC
UGG

FIGURE F 31a

2AM

TARGET CONC
IIGG

FOUND CONC
IIGG

FIGURE F 31b

2AMDNT

FOUND CONC
UGG

TARGET CONC
UGG

FIGURE F 32a

4AMDNT

TARGET CONC
UGG

FOUND CONC
UGG

FIGURE F. 32b

4AMDNT

TARGET CONC
UGG

FOUND CONC
UGG
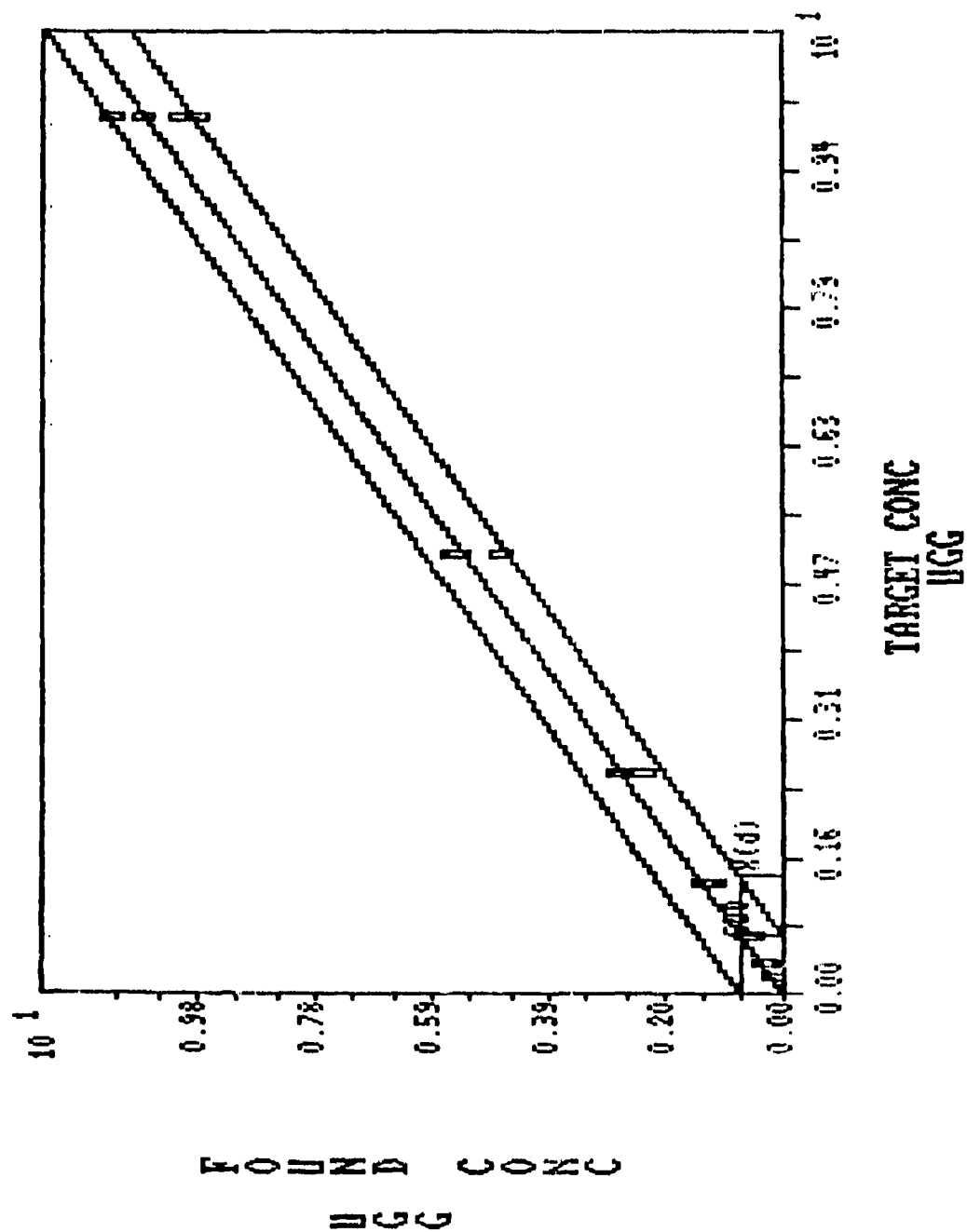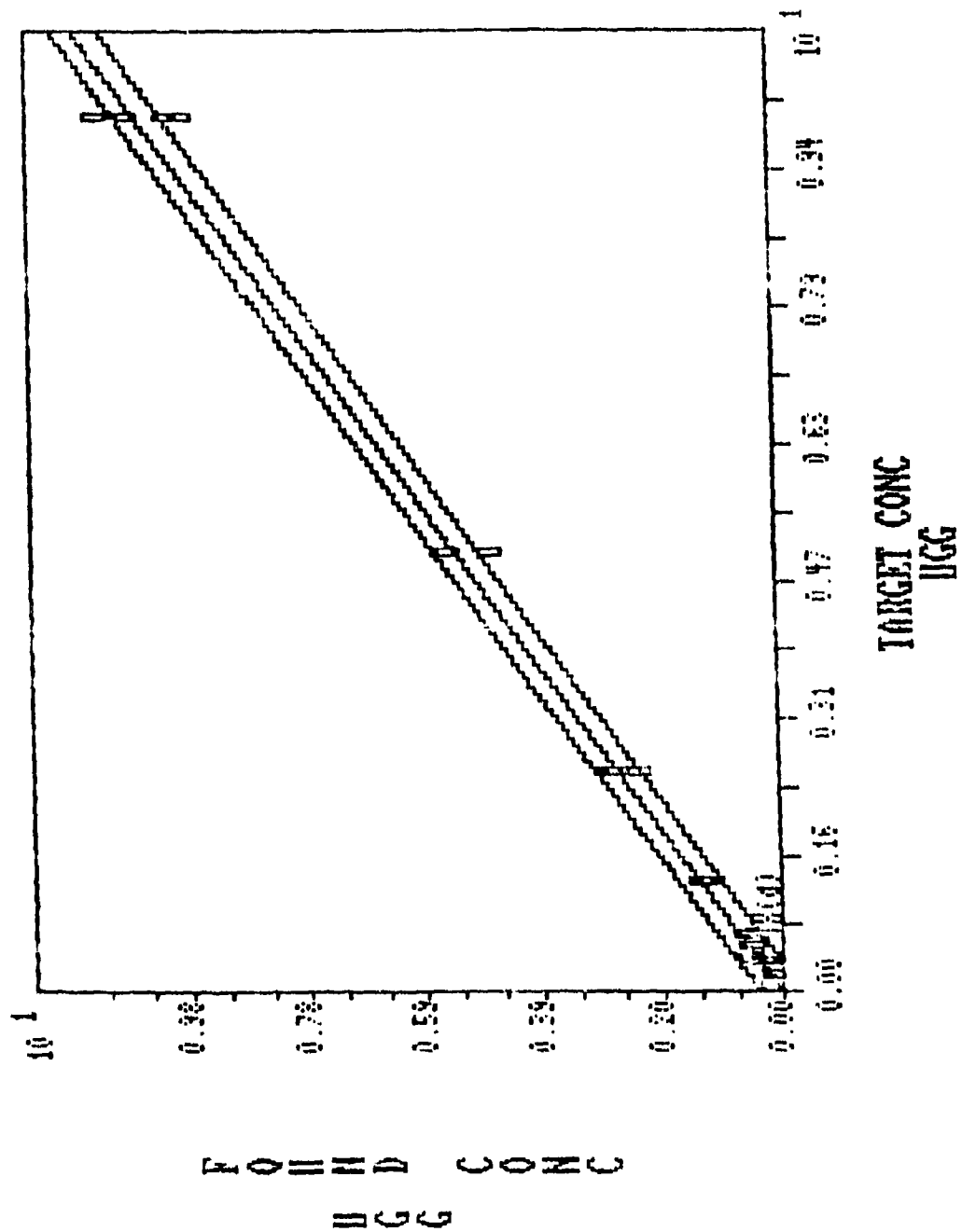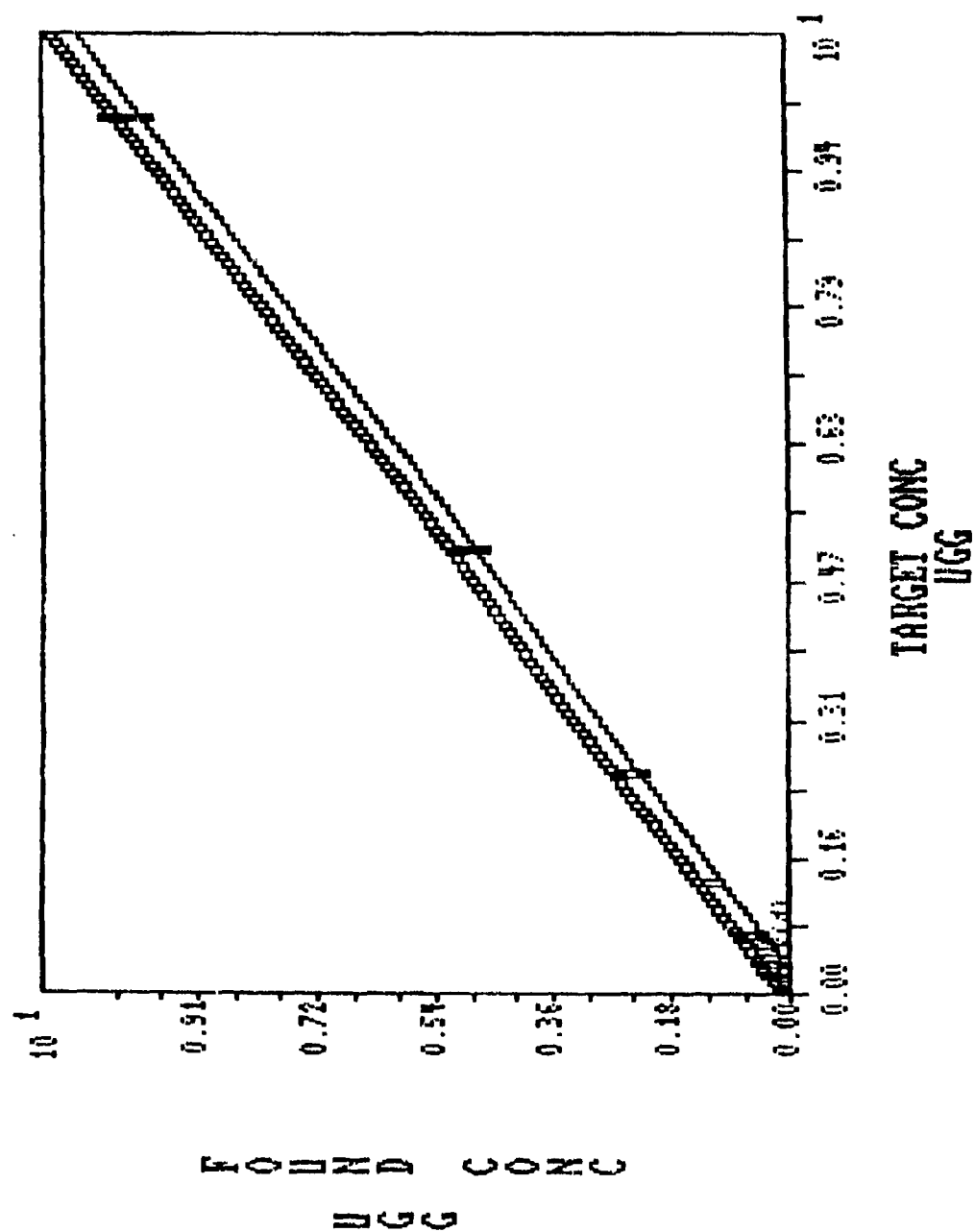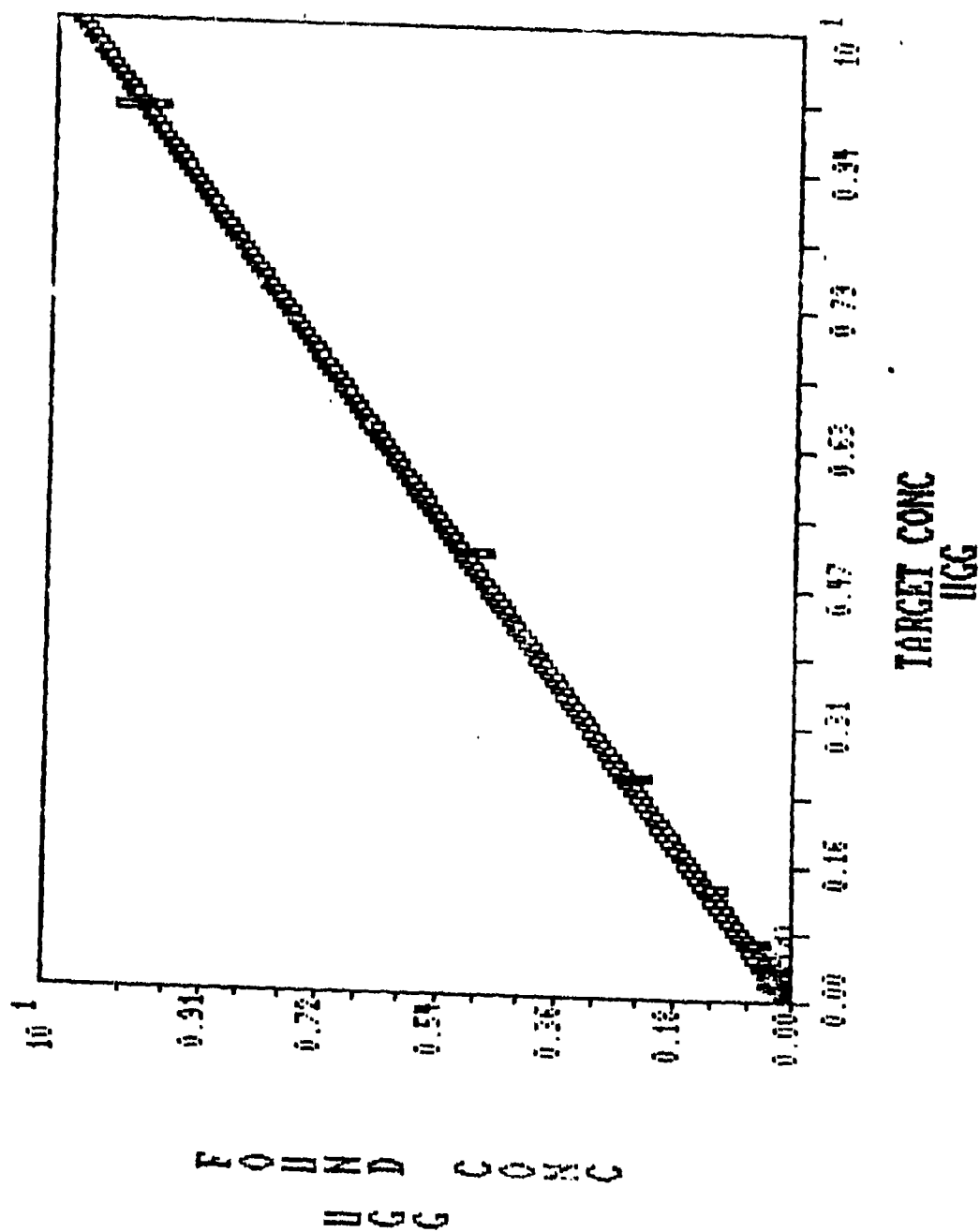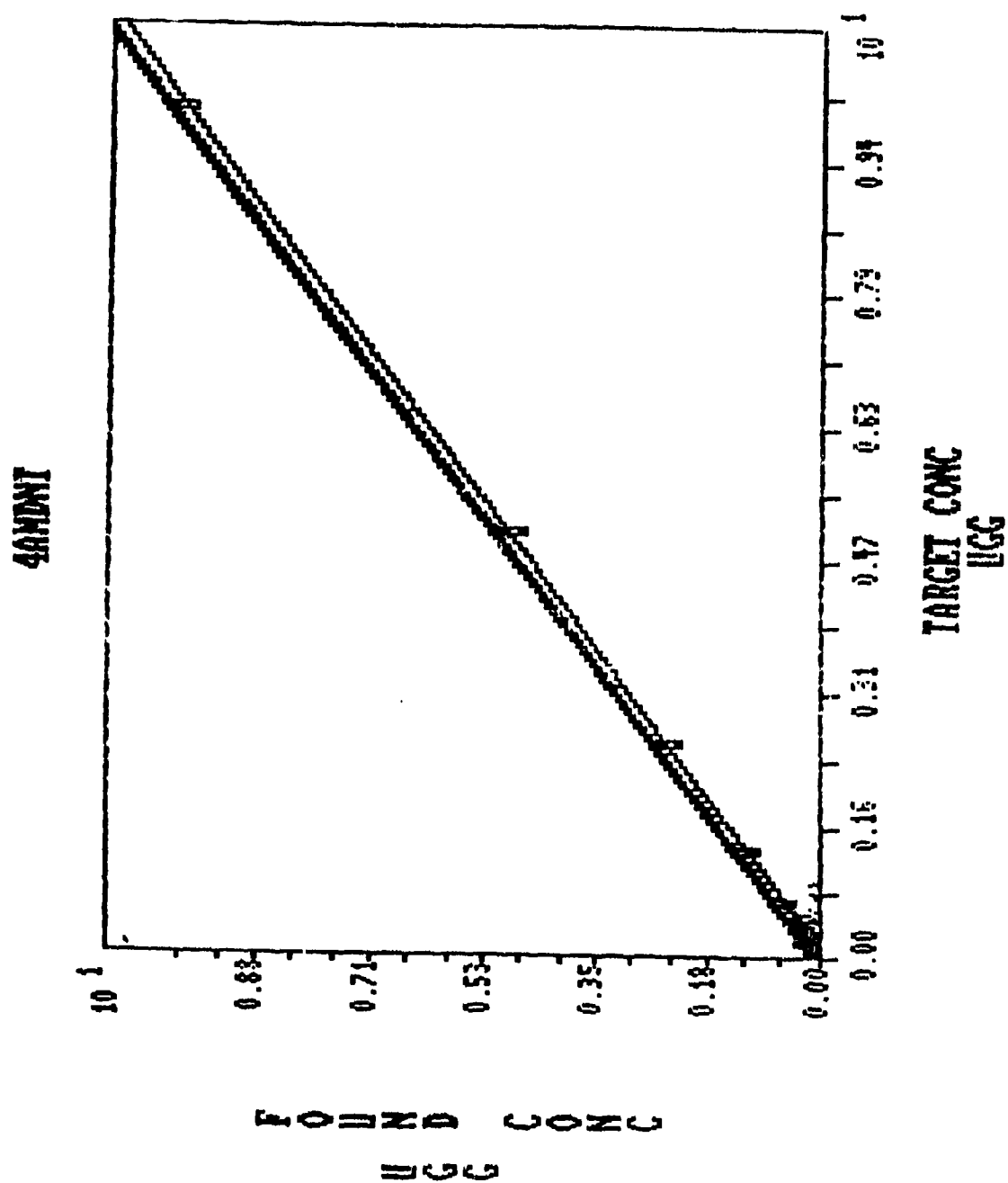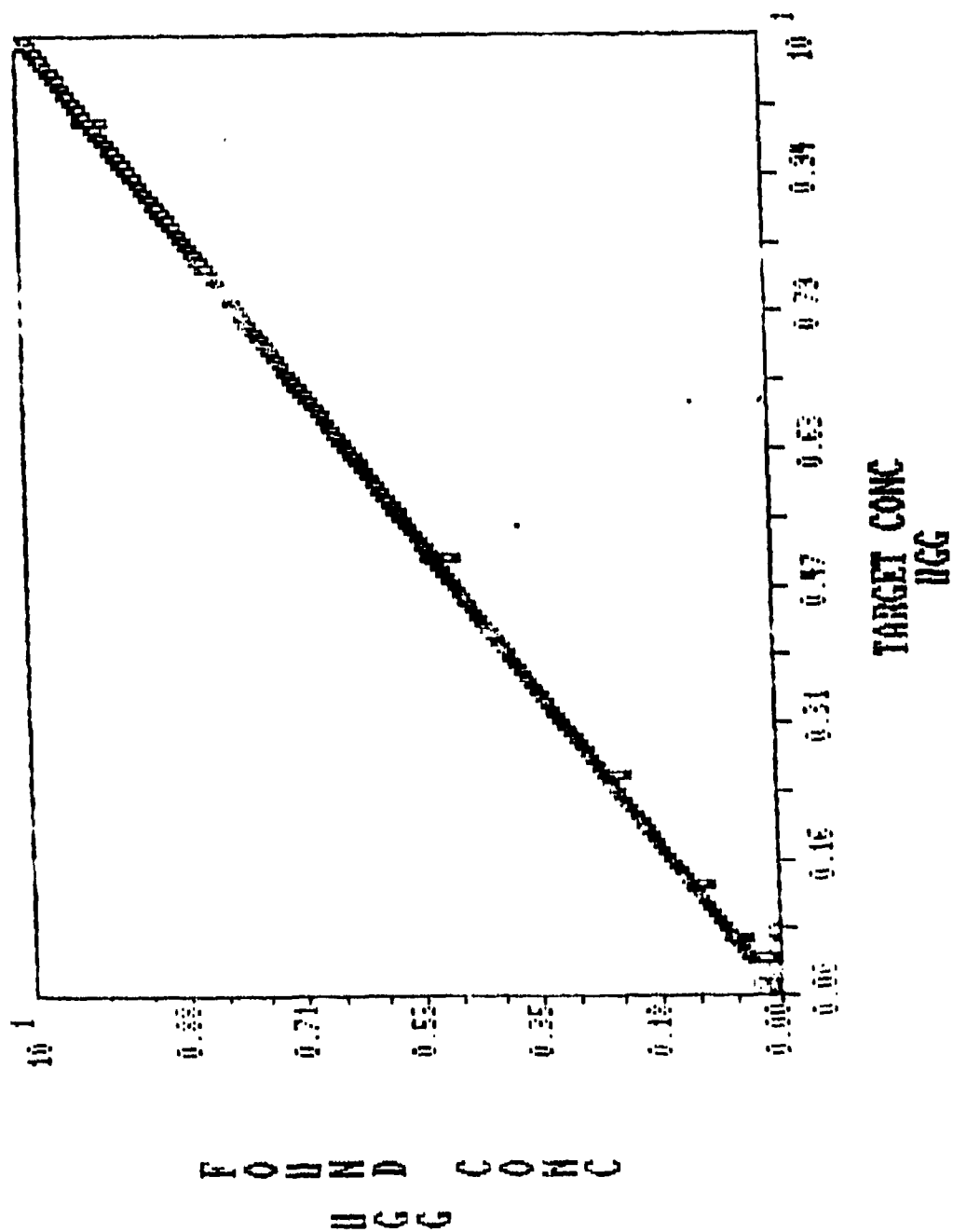
TABLE F18
CRITERION OF DETECTION WATER AND SOLVENT (mg/L)

COMPOUND	CD-R	CD-M
HMX	0.14	0.14
TNB	0.13	0.15
RDX	0.17	0.07
DNB	0.15	0.15
TNT	0.09	0.09
2,4 DNT	0.18	0.17
2,6 DNT	0.35	0.37
2-AM	0.14	0.14
4-AM	0.10	0.12

CD-R=Detection for Radford; CD-M Detection for Milan

METAL ANALYSES FOR RAAP

Concentrations of selected metals were determined for soil from RAAP site. Samples from uncontaminated, contaminated, and contaminated/fortified soils were extracted to determine total extractable Cd, Cr, Cu, Pb, and Zn levels. Duplicate 4-g air-dried samples were heated with 20 mL of 1.0 M HNO_3 for 3 h, filtered by gravity, and diluted to a 50-mL volume with ultrapure water (reverse osmosis followed by double deionization). All extracts were analyzed for metals by atomic absorption spectrophotometry (Perkin Elmer Model 3030 AA Spectrometer). Corresponding standard solutions, and blank, duplicate and split samples were also analyzed to assure quality control. Mean values of metal levels are presented in table C-1.

Table C-1. Concentrations of selected metals from Radford Army Ammunition Plant (RAAP) soils.

	Cd	Cr	Cu	Pb	Zn
	.... mg kg^{-1}				
Uncontaminated					
	0.54 ± 0.02	6.7 ± 0.1	6.0 ± 0.2	12.9 ± 0.1	67 ± 0.5
Contaminated					
	1.1 ± 0.05	10.9 ± 0.2	30 ± 3.9	183 ± 22	303 ± 44
Contaminated Fortified					
	0.95 ± 0.04	12.9 ± 0.04	23 ± 1.3	137 ± 34	294 ± 12

BLANK

APPENDIX D

RADFORD ARMY AMMUNITION PLANT

MUNITION RESIDUE DATA FROM SOIL AND LEACHATE SAMPLES

The amount of munition residue in each leachate was calculated by multiplication of the sample volume by the concentration. The amount of residue in each soil section was calculated by multiplication of the concentration of munition residue in the soil by the soil weight.

When a value of less than the criteria of detection (trace concentration) appears in tables of concentration, an "*" was entered in the corresponding amount table (concentration x leachate volume or concentration x soil weight). Zero values in the amount tables corresponded to a "none detected" (0) level in the concentration tables.

TABLE D-1.   Leachate volumes (mL) from Radford Army Ammunition Plant (RAAP) soil columns.

DAY #	14	28	42	56	70	84	98
POS#	AUG 3	AUG 17	SEP 1	SEP 15	SEP 29	OCT 13	OCT 27
				mL			
1	980	830	770	880	800	710	-
2	808	810	870	900	810	710	-
7	770	800	800	800	790	660	-
8	868	870	770	950	820	720	-
3	788	810	780	840	820	700	780
4	947	810	820	930	830	710	790
9	750	760	890	900	840	700	760
10	998	850	820	890	820	710	780
5	859	850	790	890	840	690	780
6	874	900	820	850	840	690	790
11	888	810	860	870	740	695	790
12	930	910	490	970	899	800	700
AVG.	871.67	834.17	790.00	889.17	820.75	707.92	771.25
STD. DEV.	78.22	41.72	97.89	45.18	35.75	31.45	28.48
%REL. STD. DEV.	8.97	5.00	12.39	5.08	4.36	4.44	8.97

DAY #	112	127	144	155	168	183	190
POS #	NOV 9	NOV 24	DEC 11	DEC 22	JAN 4	JAN 19	FEB 1
				mL			
1	.	.	.	.	.	.	.
2	.	.	.	.	.	.	.
7	-	.	.	..	.	.	.
8	.	.	.	.	.	.	-
3	730	730	850	630	560	695	-
4	720	710	872	660	530	730	-
9	700	740	860	660	520	730	-
10	690	780	850	650	550	750	-
5	680	440	768	600	535	720	780
6	670	730	780	620	540	510	810
11	660	720	775	620	530	710	800
12	825	500	930	770	330	970	760
AVG.	709.38	668.75	835.63	651.25	511.88	726.88	787.50
STD. DEV.	49.02	117.31	53.13	49.10	69.73	116.21	19.20
%REL. STD. DEV.	5.00	12.39	5.08	4.36	4.44	3.70	2.44

TABLE D-1. Continued...

DAY #	210	225	239	253	267	274
POS #	FEB 15	MAR 2	MAR 16	MAR 30	APR 13	APR 20

				mL		
1	.	.	.	.	.	.
2	.	.	.	"	.	.
7	.	.	.	.	.	.
8	.	.	.	.	.	.
3	.	.	.	.	.	.
4	.	.	.	.	.	.
9	..	.	.	.	.	.
10	.	.	.	.	.	.
5	750	680	710	740	725	340
6	770	700	730	760	590	300
11	800	680	700	860	740	305
12	805	670	450	1140	720	295
AVG.	781.25	682.50	647.50	875.00	693.75	310.00
STD. DEV.	22.46	10.90	114.54	159.61	60.35	17.68
%REL. STD. DEV.	2.88	1.60	17.69	18.24	8.70	5.70

Appendix D

145

TABLE D-2.   Concentrations (mg/L) of munition residues in aqueous leachates
              collected from RAAP soil columns.

              No detectable concentrations of TNB, TNT, 2,4-DNT, 2,6-DNT,
              2-amino-DNT, and 4-amino-DNT were found in any leachates
              collected from RAAP soil columns.

TABLE D-3.   Amounts (ug) of munition residues in aqueous leachates collected
              from RAAP soil columns.

              No detectable amounts of TNB, TNT, 2,4-DNT, 2,6-DNT, 2-amino-DNT,
              and 4-amino-DNT were found in any leachates collected from RAAP
              soil columns.

Appendix D

TABLE D-4.1. Concentrations (mg/kg) of munition residues in soil sections
(triplicates) from RAAP soil columns, after 0 months of leaching
(time zero).

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #s 1,2,3,5,6,8,9,10,11 (Treatment columns)							
		....		mg/kg	....		
1	AVG.	103.8	61.7	115.8	36.6	0	0
	STD. DEV.	1	7.5	0.2	0.8	0	0
	%REL. STD. DEV.	0.96	12.16	0.17	2.18	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #s 4,7,12 (Control columns)

1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

TABLE D-4.2. Concentrations (mg/kg) of munition residues in soil sections
(triplicates) from RAAP soil columns, after 3 months of leaching.

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #1		--------------------- mg/kg ---------------------					
1	AVG.	<2.4	6.61	<5.7	<5.2	<15.4	0
	STD. DEV.	-	1.18	-	-	-	0
	%REL. STD. DEV.	-	17.9	-	-	-	0
2	AVG.	<2.4	<6.1	<5.7	0	<15.4	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #2							
1	AVG.	<2.4	7.21	<5.7	0	0	0
	STD. DEV.	-	0.77	-	0	0	0
	%REL. STD. DEV.	-	10.71	-	0	0	0
2	AVG.	<2.4	<6.1	<5.7	0	<15.4	<14.6
	STD. DEV.	-	-	-	0	-	-
	%REL. STD. DEV.	-	-	-	0	-	-
3	AVG.	0	0	<5.7	0	0	0
	STD. DEV.	0	0	-	0	0	0
	%REL. STD. DEV.	0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #7 (Control)							
1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

TABLE D-4.2. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #8		--------------------			mg/kg	--------------------	
1	AVG.	<2.4	8.5	<5.7	0	0	0
	STD. DEV.	-	1	-	0	0	0
	%REL. STD. DEV.	-	11.77	-	0	0	0
2	AVG.	<2.4	<6.1	<5.7	0	<15.4	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	0	0	<15.4	0
	STD. DEV.	0	0	0	0	-	0
	%REL. STD. DEV.	0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.3.   Concentrations (mg/kg) of munition residues in soil sections
             (triplicates) from RAAP soil columns, after 6 months of leaching

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #3		-----------------			mg/kg	-------------------	
1	AVG.	<2.4	<6.1	<5.7	0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
2	AVG.	0	<6.1	<5.7	0	0	0
	STD. DEV.	0	-	-	0	0	0
	%REL. STD. DEV.	0	-	-	0	0	0
3	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #4 (Control)							
1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

COLUMN #9							
1	AVG.	<2.4	<6.1	<5.7	0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
2	AVG.	<2.4	<6.1	<5.7	0	0.	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
3	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

Appendix D   150

TABLE D-4.3. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #10		-----------------			mg/kg	--------------------	
1	AVG.	<2.4	7.84	<5.7	0	0	0
	STD. DEV.	-	1.97	-	0	0	0
	%REL. STD. DEV.	-	25.1	-	0	0	0
2	AVG.	0	<6.1	0	0	0	0
	STD. DEV.	0	-	0	0	0	0
	%REL. STD. DEV.	0	-	0	0	0	0
3	AVG.	0	0	<5.7	0	0	0
	STD. DEV.	0	0	-	0	0	0
	%REL. STD. DEV.	0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.4. Concentrations (mg/kg) of munition residues in soil sections (triplicates) from RAAP soil columns, after 9 months of leaching.

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #5		··················			mg/kg	··················	
1	AVG.	<2.4	<6.1	<5.7	.0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
2	AVG.	<2.4	<6.1	<5.7	0	<15.4	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	0	0	<15.4	0
	STD. DEV.	0	0	0	0	-	0
	%REL. STD. DEV.	0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #6							
1	AVG.	<2.4	10.44	<5.7	<5.2	<15.4	0
	STD. DEV.	-	4.33	-	-	-	0
	%REL. STD. DEV.	-	41.53	-	-	-	0
2	AVG.	<2.4	<6.1	<5.7	<5.2	<15.4	0
	STD. DEV.	-	-	-	-	-	0
	%REL. STD. DEV.	-	-	-	-	-	0
3	AVG.	0	0	0	0	<15.4	0
	STD. DEV.	0	0	0	0	-	0
	%REL. STD. DEV.	0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.4. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #11				----- mg/kg -----			
1	AVG.	<2.4	<6.1	<5.7	<5.2	<15.4	0
	STD. DEV.	-	-	-	-	-	0
	%REL. STD. DEV.	-	-	-	-	-	0
2	AVG.	<2.4	<6.1	<5.7	0	<15.4	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	<5.7	0	0	0
	STD. DEV.	0	0	-	0	0	0
	%REL. STD. DEV.	0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #12 (Control)

1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

TABLE D-4.5. Amounts (ug) of munition residues in each soil-core section
          (triplicates) from RAAP soil columns, after 0 months of leaching
          (time zero).

SAMPLE ID	TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT

Depth (inches; 2.54-cm sections)

COLUMN #s 1,2,3,5,6,8,9,10,11 (Treatment columns)

		....			ug	....	
1	AVG.	25950	15425	28950	9150	0	0
	STD. DEV.	250	1875	50	200	0	0
	%REL. STD. DEV.	0.96	12.16	0.173	2.18	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #s 4,7,12 (Control columns)

1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

TABLE D-4.6. Amounts (ug) of munition residues in each soil-core section
(triplicates) from RAAP soil columns, after 3 months of leaching.

SAMPLE ID			TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)								
COLUMN #1						ug		
1	AVG.		*	1859	*	*	*	0
	STD. DEV.		-	333.58	-	.-	-	0
	%REL. STD. DEV.		-	17.9	-	-	-	0
2	AVG.		*	*	*	0	*	0
	STD. DEV.		-	-	-	0	-	0
	%REL. STD. DEV.		-	-	-	0	-	0
3	AVG.		0	0	0	0	0	0
	STD. DEV.		0	0	0	0	0	0
	%REL. STD. DEV.		0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #2								
1	AVG.		*	2025.79	*	0	0	0
	STD. DEV.		-	217.1	-	0	0	0
	%REL. STD. DEV.		-	10.71	-	0	0	0
2	AVG.		*	*	*	0	*	*
	STD. DEV.		-	-	-	0	-	-
	%REL. STD. DEV.		-	-	-	0	-	-
3	AVG.		0	0	*	0	0	0
	STD. DEV.		0	0	-	0	0	0
	%REL. STD. DEV.		0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #7 (Control)								
1	AVG.		0	0	0	0	0	0
	STD. DEV.		0	0	0	0	0	0
	%REL. STD. DEV.		0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.6. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #8		....			ug	....	
1	AVG.	*	2390.44	*	0	0	0
	STD. DEV.	-	281.38	-	0	0	0
	%REL. STD. DEV.	-	11.77	-			
2	AVG.	*	*	*	0	*	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	0	0	*	0
	STD. DEV.	0	0	0	0	-	0
	%REL. STD. DEV.	0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.7. Amounts (ug) of munition residues in each soil-core section (triplicates) from RAAP soil columns, after 6 months of leaching.

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #3		------------------			ug	-----------------	
1	AVG.	*	*	*	0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
2	AVG.	0	*	*	0	0	0
	STD. DEV.	0	-	-	0	0	0
	%REL. STD. DEV.	0	-	-	0	0	0
3	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #4 (Control)

		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.

COLUMN #9

		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
1	AVG.	*	*	*	0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
2	AVG.	*	*	*	0	0	0
	STD. DEV.	-	-	-	0	0	0
	%REL. STD. DEV.	-	-	-	0	0	0
3	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.7. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #10		....			ug	....	
1	AVG.	*	2202.94	*	0	0	0
	STD. DEV.	-	552.88	-	0	0	0
	%REL. STD. DEV.	-	25.1	-	0	0	0
2	AVG.	0	*	0	0	0	0
	STD. DEV.	0	-	0	0	0	0
	%REL. STD. DEV.	0	-	0	0	0	0
3	AVG.	0	0	*	0	0	0
	STD. DEV.	0	0	-	0	0	0
	%REL. STD. DEV.	0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.8. Amounts (ug) of munition residues in each soil-core section
(triplicates) from RAAP soil columns, after 9 months of leaching.

SAMPLE ID			TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)								
COLUMN #5						ug		
1	AVG.		*	*	*	.0	0	0
	STD. DEV.		-	-	-	0	0	0
	%REL. STD. DEV.		-	-	-	0	0	0
2	AVG.		*	*	*	0	*	0
	STD. DEV.		-	-	-	0	-	0
	%REL. STD. DEV.		-	-	-	0	-	0
3	AVG.		0	0	0	0	*	0
	STD. DEV.		0	0	0	0	-	0
	%REL. STD. DEV.		0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #6								
1	AVG.		*	2936.38	*	*	*	0
	STD. DEV.		-	1219.53	-	-	-	0
	%REL. STD. DEV.		-	41.53	-	-	-	0
2	AVG.		*	*	*	*	*	0
	STD. DEV.		-	-	-	-	-	0
	%REL. STD. DEV.		-	-	-	-	-	0
3	AVG.		0	0	0	0	*	0
	STD. DEV.		0	0	0	0	-	0
	%REL. STD. DEV.		0	0	0	0	-	0

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

Appendix D

159

TABLE D-4.8. Continued...

SAMPLE ID		TNB	TNT	2,4-DNT	2,6-DNT	2-AM-DNT	4-AM-DNT
Depth (inches; 2.54-cm sections)							
COLUMN #11		....			ug	....	
1	AVG.	*	*	*	*	*	0
	STD. DEV.	-	-	-	-	-	0
	%REL. STD. DEV.	-	-	-	-	-	0
2	AVG.	*	*	*	0	*	0
	STD. DEV.	-	-	-	0	-	0
	%REL. STD. DEV.	-	-	-	0	-	0
3	AVG.	0	0	*	0	0	0
	STD. DEV.	0	0	-	0	0	0
	%REL. STD. DEV.	0	0	-	0	0	0

Below this depth: no detectable concentrations of munition residues.

COLUMN #12 (control)

1	AVG.	0	0	0	0	0	0
	STD. DEV.	0	0	0	0	0	0
	%REL. STD. DEV.	0	0	0	0	0	0

At all depths: no detectable concentrations of munition residues.